GENERAL SUGGESTIONS

1. Follow Directions. Before performing an experiment, read the directions through carefully. Make sure that you understand the object of the experiment. Follow the directions closely; remember that every word is significant.

2. Notebook. A careful record of each experiment performed should be kept in a substantial notebook. Record the date, the title of the experiment, and the subheadings and paragraph letters (*e.g.* a, b).

Do not duplicate the printed directions. State briefly in your own words *what you did*, *what you observed*, and *what conclusions you drew*. Answer every direct question by a definite statement. Make a simple labeled diagram of the apparatus.

3. Quantities of materials. Do not take more of the materials than the amount specified. To take more means a waste of material and of time.

4. Disposal of waste. Such things as matches, filter papers, bits of glass, and unconsumed chemicals should be thrown into the waste jars and not into the sink. If much acid or other liquids are emptied into the sinks, flush it out with plenty of water.

5. Regulate your burner. The size of the burner flame should be regulated by the stopcock. A small flame is often better than a large one for heating test tubes and beakers.

6. Neatness is essential. Make sure your apparatus is clean. After each day's work clean up your apparatus and put your desk in order.

TABLE OF THE MORE IMPORTANT ELEMENTS

with their symbols, approximate atomic weights, and valences

Element	Symbol	Approx. At. Wt.	Valence	Element	Symbol	Approx. At. Wt.	Valence
Aluminum	Al	27	III	Manganese	Mn	55	II IV
Barium	Ba	137	II	Mercury	Hg	200	I II
Bromine	Br	80	I	Nickel	Ni	58.7	II
Calcium	Ca	40	II	Nitrogen	N	14	III V
Carbon	C	12	IV	Oxygen	O	16	II
Chlorine	Cl	35.5	I	Phosphorus	P	31	III V
Chromium	Cr	52	II III IV	Platinum	Pt	195	IV
Copper	Cu	63.6	I II	Potassium	K	39	I
Fluorine	F	19	I	Radium	Ra	226	II
Gold	Au	197	I III	Silicon	Si	28	IV
Helium	He	4	——	Silver	Ag	108	I
Hydrogen	H	1	I	Sodium	Na	23	I
Iodine	I	127	I	Sulfur	S	32	II IV VI
Iron	Fe	56	II III	Tin	Sn	119	II IV
Lead	Pb	207	II IV	Tungsten	W	184	VI
Magnesium	Mg	24	II	Zinc	Zn	65	II

COMMON UNITS IN THE METRIC AND ENGLISH SYSTEMS

10 millimeters (mm.) = 1 centimeter (cm.)
100 centimeters = 1 meter (m.)
1000 cubic centimeters (cc.) = 1 liter (l.)
10 milligrams (mg.) = 1 centigram (cg.)
100 centigrams = 1 gram (g.)
1000 grams = 1 kilogram (kg.)

1 cc. of water at 4° C. weighs 1 gram
2.54 centimeters = 1 inch
1 meter = 39.37 inches
1 liter = 1.06 quarts
1 gram = 15.4 grains
28.35 grams = 1 ounce
1 kilogram = 2.20 pounds
1 pint of water weighs about 1 pound


1861

Goddard Seminary

LABORATORY EXPERIMENTS IN CHEMISTRY

For Red Fire

3/4 g $KClO_3$
4 1/2 g Strontium nitrate
1 1/2 g Powdered Shellac. (Orange)

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LABORATORY EXPERIMENTS IN CHEMISTRY

TO ACCOMPANY

BLACK AND CONANT'S "PRACTICAL CHEMISTRY"

BY

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PREFACE

LABORATORY work in any science is fundamental and the study of a text book merely extends and organizes the information which the student gets in the laboratory. The experiments which the student does with his own hands should make real and concrete the fundamental principles of the science. It is also essential for him to perform certain experiments which indicate how these facts and principles are utilized at home and in the commercial and industrial life of the community.

The directions for these experiments are at first very full and detailed, but are gradually abbreviated so as to leave more and more to the student's own initiative and imagination. It is hoped that the directions are so clearly stated and illustrated that the teacher will be largely freed from the necessity of explaining the details of manipulation. Frequent questions (in italics) have been inserted in the directions to focus the attention of the student on the important facts to be observed.

One of the aims of all laboratory work is to arouse the student's spirit and enthusiasm for finding out things by experiment. To encourage this spirit numerous questions, problems, and experiments have been suggested as *optional work.* These may well be used to give elasticity and variety to the laboratory work as well as to emphasize the close connection between chemistry in the school laboratory and chemistry in the household, in the factory, or on the farm.

The instructor is expected to select those experiments which meet the needs of his class. Probably *an average class of beginners in chemistry cannot perform successfully more than*

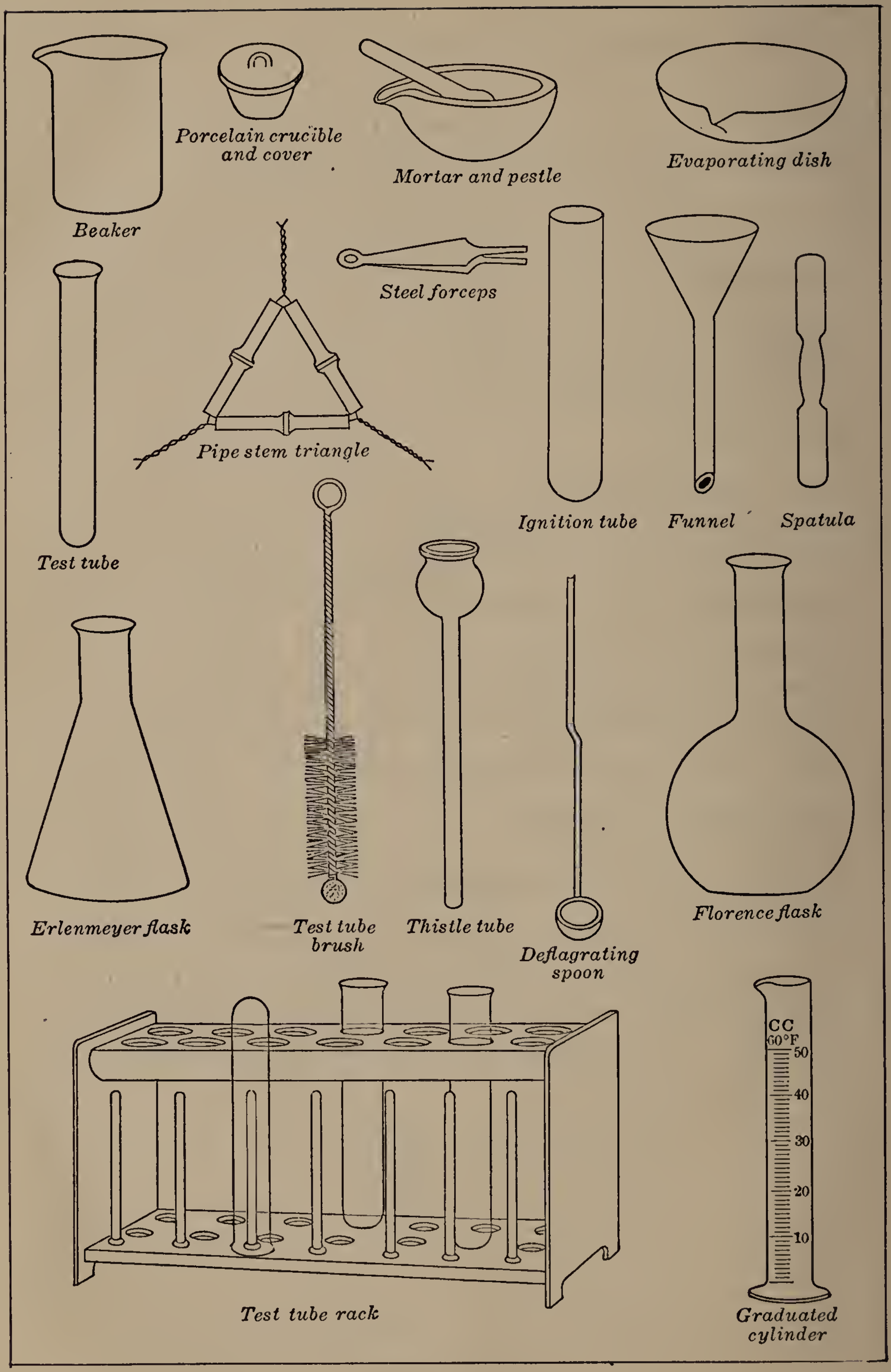
Camp Sheri. From Library Eugene Averill

forty of these experiments. It is far better to have a small number of experiments carefully done, well written up, and thoroughly understood, than many experiments carelessly rushed through.

In the present laboratory manual there is little material which is either new or original; but special attention has been paid to selecting suitable experiments and to wording the directions. In this work the author has found the following books especially useful and suggestive: Elementary Studies in Chemistry by *Joseph Torrey,* School Chemistry by *Wilson and Hadley,* Laboratory Study of Chemistry by *Smith and Mess,* and Laboratory Outline of General Chemistry by *Alexander Smith;* also the Syllabus in Chemistry for Secondary Schools published by the Regents of New York State, and the printed laboratory directions used at Harvard College and at the Massachusetts Institute of Technology. The author is also greatly indebted to Professor James Bryant Conant, Dr. Norris F. Hall, and Mr. Bancroft Beatley, all of Harvard University, for their aid in the correction of the proof sheets.

The apparatus required for the essential experiments is simple and the chemicals are inexpensive (see lists in Appendix). Those experiments marked in the table of contents with a star (*) deal with fundamental facts and should be in any first-year course.

TABLE OF CONTENTS


Beaker
Porcelain crucible and cover
Mortar and pestle
Evaporating dish
Test tube
Pipe stem triangle
Steel forceps
Ignition tube
Funnel
Spatula
Erlenmeyer flask
Test tube brush
Thistle tube
Deflagrating spoon
Florence flask
Test tube rack
CC
60°F
50
40
30
20
10
Graduated cylinder

LABORATORY EXPERIMENTS IN CHEMISTRY

HINTS ABOUT LABORATORY MANIPULATION

Check up your apparatus. — Make sure that your outfit is complete and in good condition. On the opposite page you will find the names and pictures of some of the less familiar pieces of apparatus. Keep the articles clean and arranged in an orderly fashion.

Bunsen burner. — In a laboratory provided with gas the Bunsen burner (Fig. 1) is almost indispensable as a source of heat. When the air holes at the base of the burner are open, air is drawn in and mixes with the gas. It should burn with a clear, blue flame (Fig. 1); the size of the flame can be controlled by adjusting the stopcock. When the holes at the base of the burner tube are closed, a yellow luminous flame results.

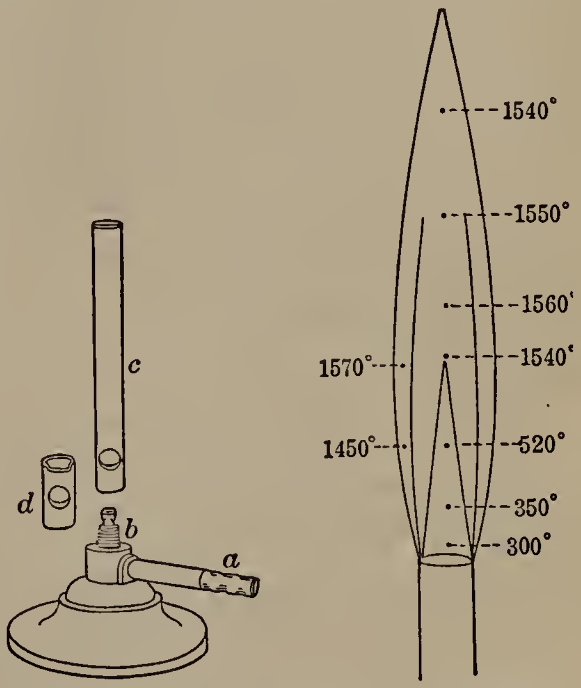


Fig. 1. **Bunsen burner and flame.**

Figures indicate probable temperatures (°C.).

In lighting such a burner, never hold the match over it while turning on the gas, but turn on the gas and then apply the match. By the former method it will probably "snap back", that is, burn at the base inside the tube. In this condition the burner gives off a disagreeable gas and becomes extremely hot. Turn off a "snapped" lamp and relight properly.

Glass tubing. **To cut** a piece of small glass tubing, lay it on the table and make a scratch with a triangular file at the point where it is to be cut. Next take the tube in both hands, placing the thumbs on the side of the tube opposite the scratch. Then press the thumbs against the tube and pull the ends of the tube towards you (Fig. 2).

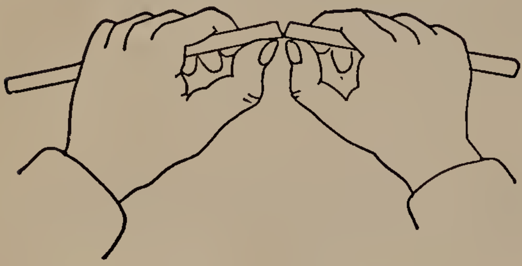


Fig. 2. How to break glass tubing.

To bend glass tubing, rotate it in a flat flame, such as that furnished by the **wing-top** on the Bunsen burner (Fig. 3), until soft and then take it out of the flame and gradually bend it into the desired shape. In order to make a good bend (Fig. 4) the tube must be heated evenly over a portion of the tube at least 5 cm. in length. Remember hot glass placed on a cold table is almost sure to break.

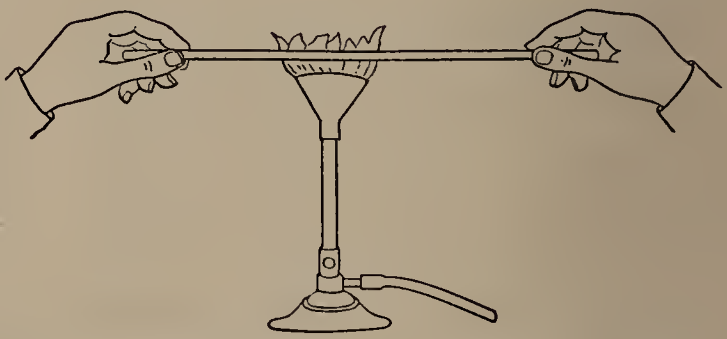


Fig. 3. Heating glass tubing in a flat flame.

Before glass tubes are used for any purpose the sharp edges must be rounded by being heated near the top of a Bunsen flame. This is called "**fire-polishing**" the ends of the tube.

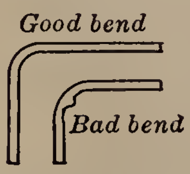


Fig. 4. A good and a bad bend.

Setting up apparatus. In putting together apparatus it is often necessary to push a glass tube through a stopper. It is a good plan to lubricate the glass tubing with water, and to push the tube in slowly with a slight twisting motion. It is also very important to leave the joints tight. To test apparatus for gas-tightness either compress the air in it by blowing or draw some air out by suction. Generally some part of the apparatus is under water and so the level of the water can be made to rise or fall in a tube. If there is a leak, this level will change slowly when the opening where the lips were applied is closed. Don't try to plug up holes with matches or wax but *set the apparatus up right.*

Heating apparatus. Wipe dry the outside of every glass or porcelain vessel before heating it. In heating a test tube containing a liquid, hold it in an inclined position with some kind of holder and keep it moving in the flame, taking care not to let the flame touch the glass above the liquid. **Don't try to boil a liquid in a flask or beaker with a direct flame.** Use the wire gauze with asbestos center. Turn the flame down until it is just the size of the beaker, boiling will begin much sooner.

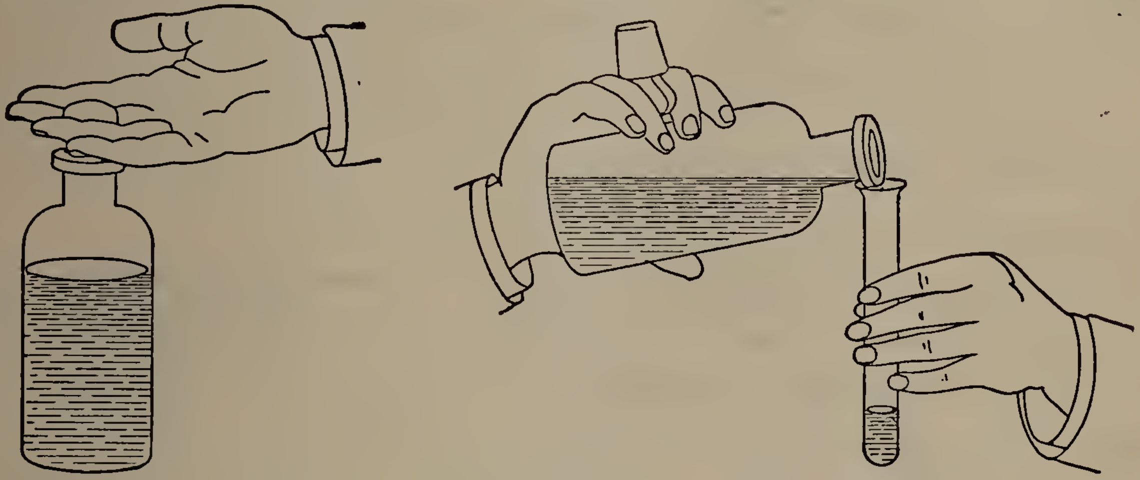


Fig. 5. Proper way to hold a stopper while pouring.

Porcelain dishes are used chiefly in evaporating and should be heated over asbestos. A porcelain crucible may be supported on the pipestem triangle and heated directly provided it is warmed up gradually and cooled off slowly.

Handling materials. Don't try to *pour solids* out of a bottle. Use some kind of spoon or ladle. Don't weigh materials directly on the scale pans, use a piece of creased paper. Don't use more material than required and don't pour back excess material into the reagent bottle.

In pouring liquids from a bottle some care must be taken to prevent the liquid from running down the side of the bottle.

Place the neck of the bottle lightly against the rim of the vessel into which the liquid is being poured. *The stopper must never be laid down on the desk.* Hold it between the fingers as shown in figure 5.

To filter a liquid first fold the filter paper in quarters and then open it up so as to form a cone (Fig. 6). Fit the cone into a funnel of such size that the paper does not quite reach the top and wet it thoroughly with water. The filter is now ready for use.

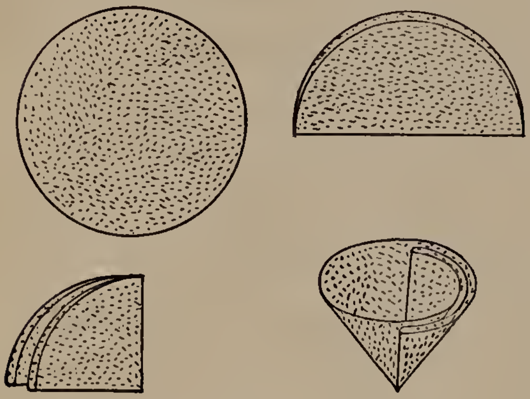


Fig. 6. Folding a filter paper.

Stirring rods should be fire-polished. They are very convenient in pouring liquids. The liquid may easily be made to flow down the rod (Fig. 7). A stirring rod should also be used in testing with litmus paper. Put a drop of the liquid to be tested on the paper, don't dip the paper into the liquid.

Weighing and counting weights. In weighing out the materials for an experiment, use the platform or trip scales (Fig. 8). If these are properly handed, they are good to one-tenth of a gram (0.1 g.). Remember the thing to be weighed is placed on the *left-hand* pan and the weights on the *right-hand* pan; also remember to use the slider for small weights.

Fig. 7. Pouring a liquid down a glass rod.

For **quantitative experiments** use the horn pan balance (Fig. 9) which is sensitive to one-hundredth of a gram (0.01 g.). A chemical balance (Fig. 10) is usually provided with agate bearings and inclosed in a glass case. Examine the box of weights and see how they are arranged and marked. Notice that the heavier weights (one gram and above) are made of brass and that the fractional weights are of German silver or aluminum and marked in milligrams. Thus 500 milligrams is 0.5 grams and 10 milligrams is 0.01 grams.

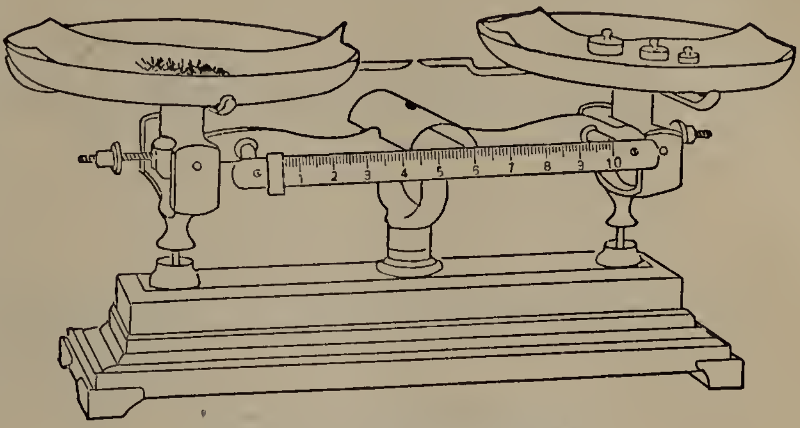


Fig. 8. Platform scales.

In weighing first see that your balance swings evenly, that is, that the pointer swings an equal distance to the right and left. If it does not, adjust it either by means of a set screw or by adding counterbalance. Place the object to be weighed on the left-hand pan and the weights on the right. First try a weight which you think is a little heavier than the object; remove it if too heavy and try smaller ones in order. Never handle the weights with your fingers; always use the forceps. Count up the weights on the pan and write down the total at once. *Experience shows that many mistakes are made by miscounting the weights.* Check this result by noting those absent from the box. Beginning with the largest weight, return them to the box and again count them as you replace them.

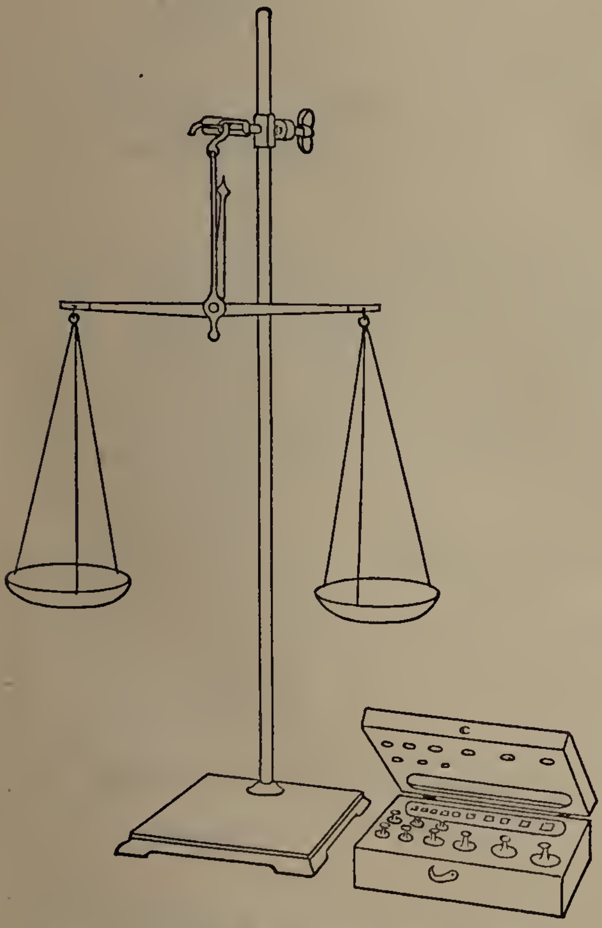


Fig. 9. Horn pan balance and set of weights.

Do not wait for a balance to come to rest. A good balance will continue to swing for a long time. Place just enough weights on the pan so that the pointer swings an equal distance to the right and left.

Measuring volumes. A graduated glass cylinder (Fig. 11) is commonly used to measure the volume of a liquid. Care must be taken to keep the eye on a level with the surface of the liquid. Get familiar with the scale markings and their value. The eye should follow the scale from the zero end. It will also be noticed that the surface of water is not flat but curved. Such a curved surface is called a **meniscus.** With a concave meniscus, such as occurs on water, the position of the lowest point of the lowest curve against the scale is to be taken as the reading (Fig. 12). In measuring small volumes use a smaller graduated cylinder, a conical graduate or a graduated test tube.

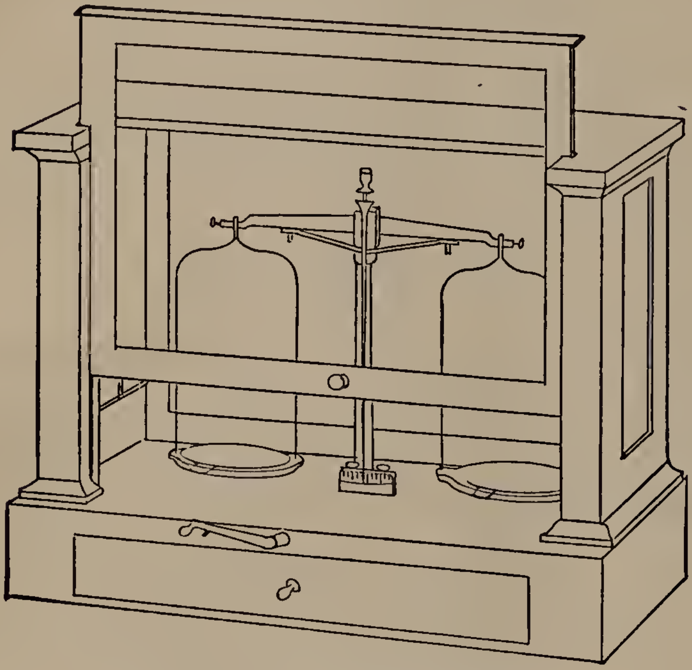


Fig. 10. Chemical balance.

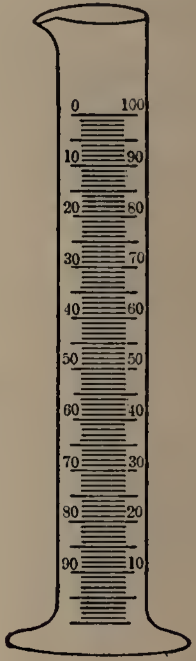


Fig. 11. Graduated glass cylinder.

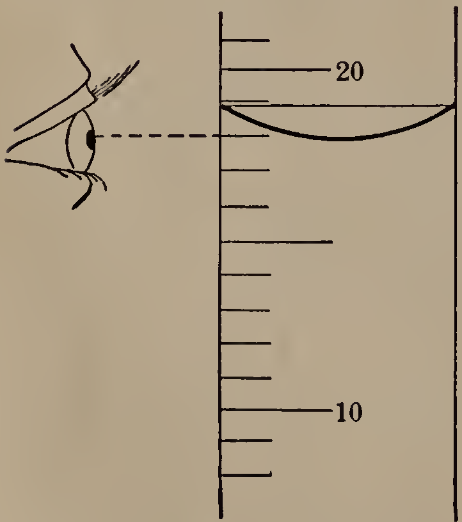


Fig. 12. Reading the meniscus of water.

HOW TO MAKE NOTEBOOK DRAWINGS

Don't try to make a picture of your apparatus, but make a **sectional drawing.** Imagine the apparatus to be cut by a vertical plane; suppose the sheet of paper to be placed in the position of this plane. Then draw the lines where the paper would cut the intersected apparatus. Figure 13 shows a sectional drawing of the apparatus used to make oxygen. It is also useful for review purposes to label the drawing. Unless you are unusually skillful in free hand drawing you had better use a ruler.

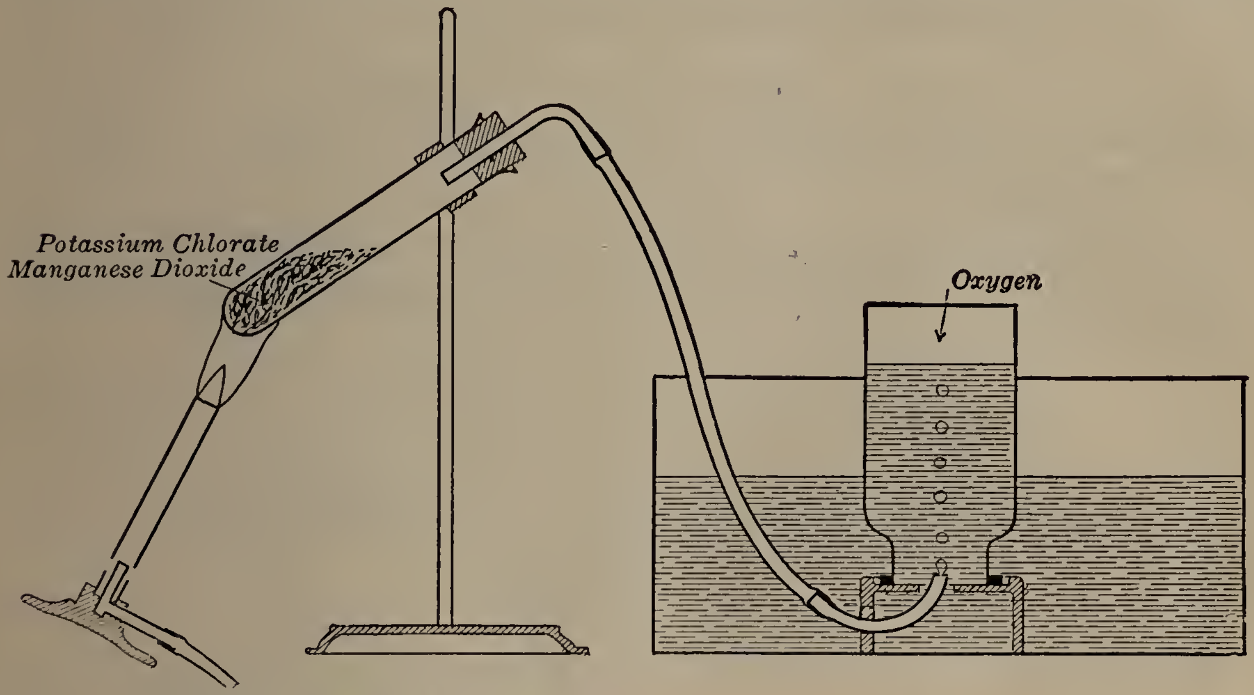


Fig. 13. Sectional drawing of oxygen-generating apparatus.

Jan. 24,

EXPERIMENT 1

Changes in Substances Heated in Air

Apparatus. — Forceps. Bunsen burner. Iron spoon.
Materials. — Copper wire (#24). Magnesium ribbon (6 cm.). Granulated sugar.

a. Examine a piece of bright copper wire and record those characteristic properties which enable one to recognize it as copper. Hold the wire with forceps in the tip of the flame of a Bunsen burner (Fig. 14) until you are sure that longer heating will produce no further change. Cool and compare the surface material with a piece that has not been heated. *Is the copper changed?*

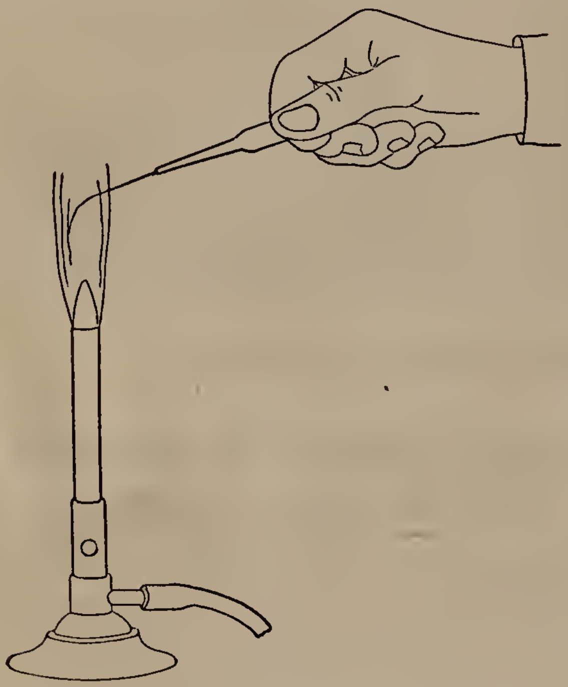


Fig. 14. **Heating wire in Bunsen flame.**

Fig. 15. **Heating sugar in an iron spoon.**

Note. Always state the evidence on which you base your conclusions.

b. Repeat the experiment (**a**), using a piece of magnesium ribbon. *Record definite properties of magnesium and of the product,* such as: color, appearance (metallic or otherwise), and pliability or brittleness.

c. Heat a bit of sugar (size of a pea) in an iron spoon as shown in figure 15. Note any flame, smoke, or odor produced. *Record the change in appearance and the relative amount of the residue. Identify it by its properties.*

Optional work.* Heat a platinum wire and compare the resulting product with the original wire. *Is it changed?* In the same way examine some tin (foil or granulated), salt, and sulfur, and record all the properties of each which you can easily determine. Heat each substance separately in the air and note what happens as well as the properties of the products.

Are these substances changed by heat? How do you know? What is the most marked difference between the behavior of sulfur and that of all the other substances? Is the sulfur annihilated?

EXPERIMENT 2

CHANGE IN WEIGHT IN A METAL HEATED IN AIR

Apparatus. — Porcelain crucible. Horn pan balance and weights. Ring stand. Pipe-stem triangle. Bunsen burner.
Materials. — Copper wire (#30).

CAUTION. Never place chemicals of any sort directly upon the scale pans. Never weigh an object while it is hot.

a. Weigh a clean, dry porcelain crucible (without cover) to the nearest centigram (0.01 g.). Wind about 5 meters of copper wire (#30) around a pencil; place the coil in the crucible and weigh the whole as before. Support the crucible on a pipe-stem triangle (Fig. 16) and heat, at first gently and then with

---

* Apparatus and material needed for the **optional work** are *not* included in the list at the beginning of the experiment.

the full Bunsen flame, for 15 minutes. The flame should be adjusted so that the crucible is just above the tip of the inner blue cone. For the purposes of this experiment it is not necessary that the action be complete.

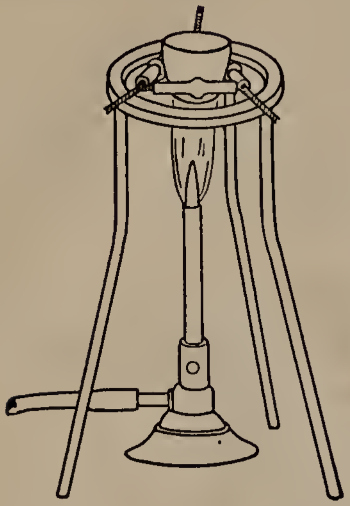


Fig. 16. Heating a crucible on a pipe-stem triangle.

b. To cool the crucible, gradually lower the flame and finally remove it, allowing the crucible to cool on the triangle until it can be held in the hand.

Weigh the crucible again.

Has there been a loss or a gain in weight? To what is the difference in weight probably due?

Record your weights in tabular form as follows:

Wt. of crucible + copper . . . . g.
Wt. of crucible empty . . . . g.
Wt. of copper taken . . . . g.
Wt. of crucible + copper after heating . . . . g.
Wt. of crucible empty . . . . g.
Wt. of copper after heating . . . . g.
Change in wt. of copper . . . . g.

Optional work. Repeat the experiment, using instead of copper 2 or 3 g. of powdered iron or granulated tin.

Jan 27.

EXPERIMENT 3

SEPARATING A MIXTURE INTO ITS COMPONENTS

Apparatus. — Test tubes and corks to fit. Funnel. Filter papers. Crystallizing dish. Small flask. Evaporating dish. Ring stand.
Materials. — Black gunpowder. Carbon disulfide.

a. Examine some black gunpowder. Although it is a mixture of three quite different substances, yet they are so carefully put together that they are not easily discernible. However, we can separate the various components by using suitable solvents. Carbon disulfide will dissolve the sulfur, water will dissolve the niter or saltpeter, and the charcoal, which is insoluble in both of these solvents, is left behind.

b. Put about 5 g. of gunpowder into a test tube to which a cork has been fitted. Pour upon the gunpowder 10 cc. of carbon disulfide, replace the cork, and shake the test tube for 5 minutes. Allow it to settle, pour off the clear liquid through a dry filter (Fig. 17) into a beaker or crystallizing dish, and set it aside where no flame will get at it.

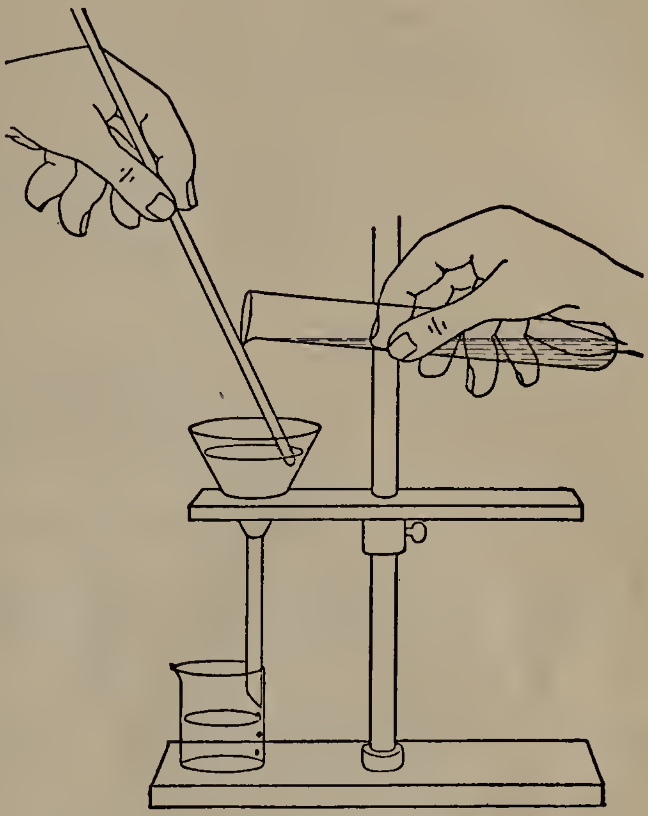


Fig. 17. Separating a mixture by filtration.

Caution. Carbon disulfide is very inflammable and must never be handled near a flame.

The carbon disulfide will soon evaporate and leave crystals of **sulfur.**

c. Spread the black residue left in the test tube on a filter paper to dry. When it has dried, transfer the residue to a small flask. Pour 20 cc. of hot water upon it and shake for 5 minutes. The saltpeter is dissolved, and the solution may be filtered into a clean evaporating dish. By heating the filtrate the water is evaporated, leaving a white residue, the **saltpeter** or **niter.**

d. The black residue left on the filter paper is the third component of gunpowder, **charcoal** or carbon.

What property of each of the three components of gunpowder was made use of in this separation?

Optional work. Make mixtures of the following substances and then separate the components by means of a suitable solvent:

(1) Powdered chalk and common salt.
(2) Powdered zinc and sulfur.
(3) Sand and sugar.

$KClO_3$ Jan. 25

EXPERIMENT 4

DECOMPOSITION OF A COMPOUND

Apparatus. — Ignition tube (hard-glass test tube) with 1-hole rubber stopper. Ring stand and clamp. Pneumatic trough. Test tubes. Glass tubing. Bunsen burner. Wing-top.

Materials. — Mercuric oxide. Splint.

a. Place about 2 or 3 g. of the red powder (mercuric oxide) in the bottom of a clean ignition tube (hard-glass test tube).

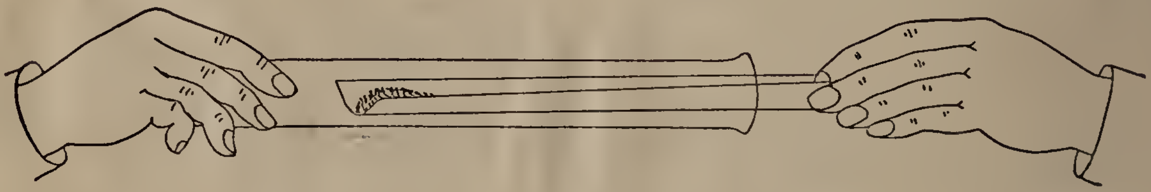


Fig. 18. Sliding a powder into a test tube.

This can be done by putting the powder near the end of a narrow strip of folded paper and sliding it carefully into the tube as shown in figure 18. By inclining the tube and gently tapping the paper, the powder will be deposited in the bottom of the tube, leaving the sides of the tube perfectly clean.

Now fit the ignition tube with a one-hole rubber stopper carrying a delivery tube which is arranged as shown in figure 19. Fill a test tube with water and invert it over the end of the delivery tube in a dish of water (pneumatic trough).

b. Warm the ignition tube very gradually by holding the burner in the hand and waving the flame along the tube. Very soon bubbles of gas will be seen coming from the end of the delivery tube.

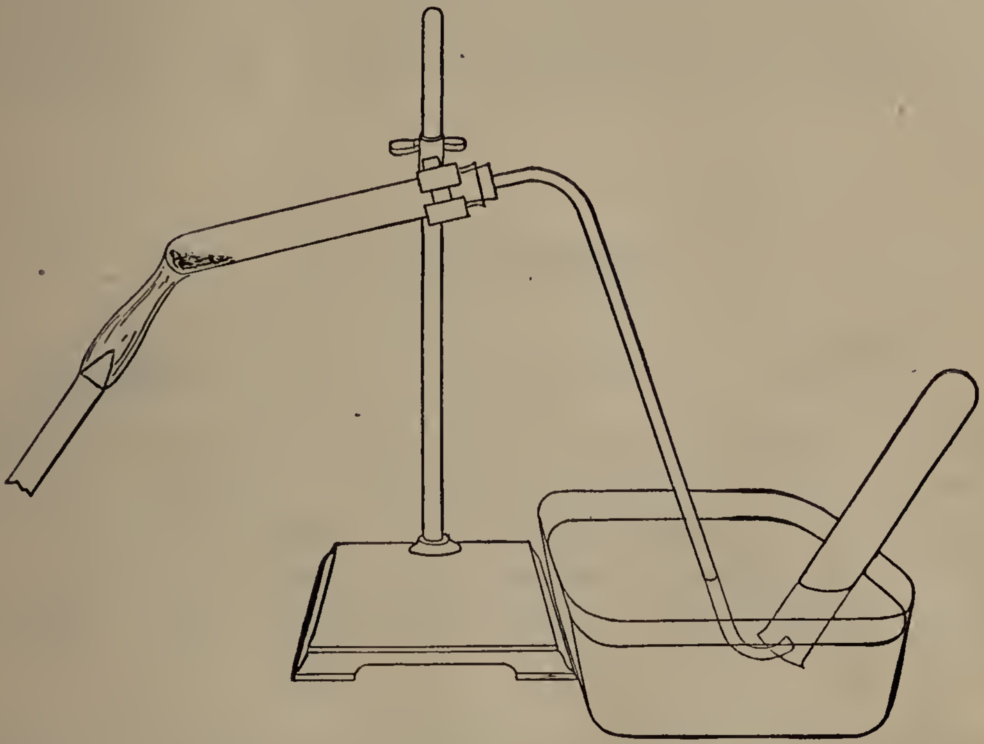


Fig. 19. Decomposing mercuric oxide.

What is the cause of this bubbling?

When half a test tube of gas has been collected, remove the test tube, invert it, and test the gas by thrusting into it a glowing (not flaming) splint. *Is the gas air?*

Caution. Never allow the ignition tube to cool while it is connected with a delivery tube which has its free end under water. *Explain what would happen.*

c. Continue heating the ignition tube until the powder is all decomposed. Collect a second test tube of gas and test with a glowing splint. This gas is oxygen. *What is the difference in the behavior of the glowing splint in the two test tubes?*

d. Scrape off with a splint some of the substance left on the side of the ignition tube. *What is it?*

Into what two substances has the red powder been decomposed?

Optional work. Place about 1 cc. of lead dioxide in an ignition tube and fasten the tube upright in the clamp on the ring stand. Heat at first gently and then strongly with the Bunsen flame. Test for oxygen by introducing a glowing splint. After removing the flame, examine the residue in the tube. This new substance is **litharge.**

EXPERIMENT 5

OXYGEN — PREPARATION AND PROPERTIES

Apparatus. — Test tube and 1-hole stopper. Ring stand with clamp. Bunsen burner. 4 wide-mouth bottles. 4 glass squares. Pneumatic trough. Deflagrating spoon. Glass tubing.

Materials. — Potassium chlorate. Manganese dioxide. Splint. Splinter of charcoal. Copper wire (15 cm. #18). Sulfur. Red phosphorus. Asbestos paper.

I. Preparation. Mix on paper about 10 g. of potassium chlorate and about one-third the bulk of manganese dioxide. Slide the mixture into a test tube, clamp it horizontally, and tap it so as to distribute the mixture along the side of the lower half of the tube, leaving space above the mixture to the end of the tube. Insert the rubber stopper carrying the delivery tube as shown in figure 20. Fill the pneumatic trough with enough water to cover the support for the bottles. Fill 4 wide-mouth bottles with water, cover them with glass plates, and invert them in the trough.

Use a small Bunsen flame to heat the tube, keep the flame moving, and remove the flame when the gas comes too fast. Heat slowly. *Will the gas which passes over at first be pure oxygen?* Now fill the bottles with oxygen one by one, covering each when full with a glass plate, and setting it mouth upwards on the table. After the fourth bottle is filled, remove the delivery tube from the water and allow the test tube to cool. The residue in the test tube may be easily removed with warm water.

II. Properties. *a.* Use one bottle of the gas to determine such properties as color, odor, and solubility, and then thrust in a glowing splint. (Sometimes a slight cloud is present when oxygen is made from potassium chlorate, due to the presence of an impurity.) The solubility can be inferred from the method of collecting the gas.

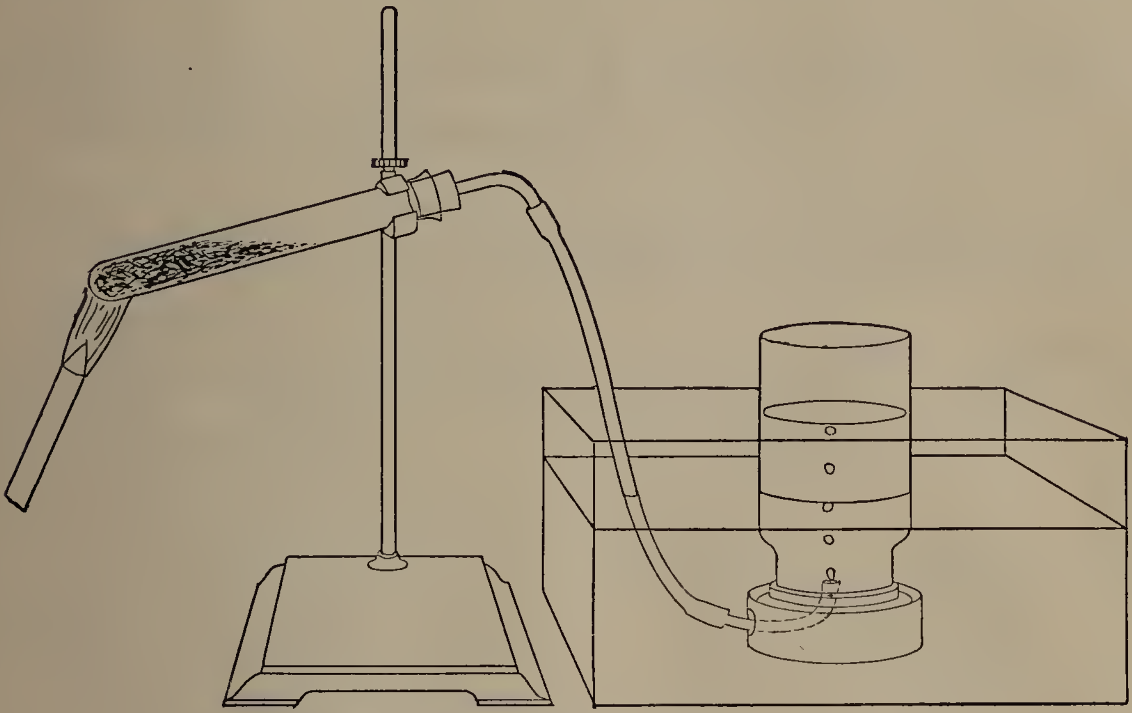


Fig. 20. **Decomposing potassium chlorate.**

b. Charcoal. Wind one end of a piece of wire around a thin splinter of charcoal. Heat the charcoal until it glows brightly and immediately lower it into a bottle of oxygen. *Compare the combustion of charcoal in oxygen with that in air. Of what does the gas produced probably consist?*

c. Sulfur. Line a combustion or **deflagrating spoon** with asbestos paper and put in it about 0.5 cc. of sulfur. Ignite the sulfur in the Bunsen flame. Observe the flame for a moment and then lower the spoon into a bottle of oxygen, keeping the mouth of the bottle closed as far as possible with the glass plate. *Compare the flame of sulfur burning in oxygen with that in air. Is it finally extinguished? If so, why?* Very cautiously smell the contents left in the bottle. *What is it?*

d. Phosphorus. Clean and reline the spoon with asbestos. Place in it a bit of red phosphorus (size of small pea), ignite and lower it into another bottle of oxygen. *Compare the burning as before. Is the product a gas, or does it consist of fine solid particles?*

Caution. Before putting the spoon away, clean it by heating it to redness for two minutes to burn up all the phosphorus.

Record the properties of oxygen which you have observed in this experiment and its most striking characteristic.

Optional work. Prepare oxygen from sodium peroxide (a white solid containing 41 per cent of oxygen). This can be conveniently done by merely dropping water on the peroxide in the apparatus shown in figure 21. Put about 5 g. of sodium peroxide in the bottom of the bottle *B* and partly fill the funnel *F* with warm water. Open the pinchcock *P* a little from time to time so that the water will fall upon the peroxide. Always keep a little water in the funnel. Collect the oxygen as before.

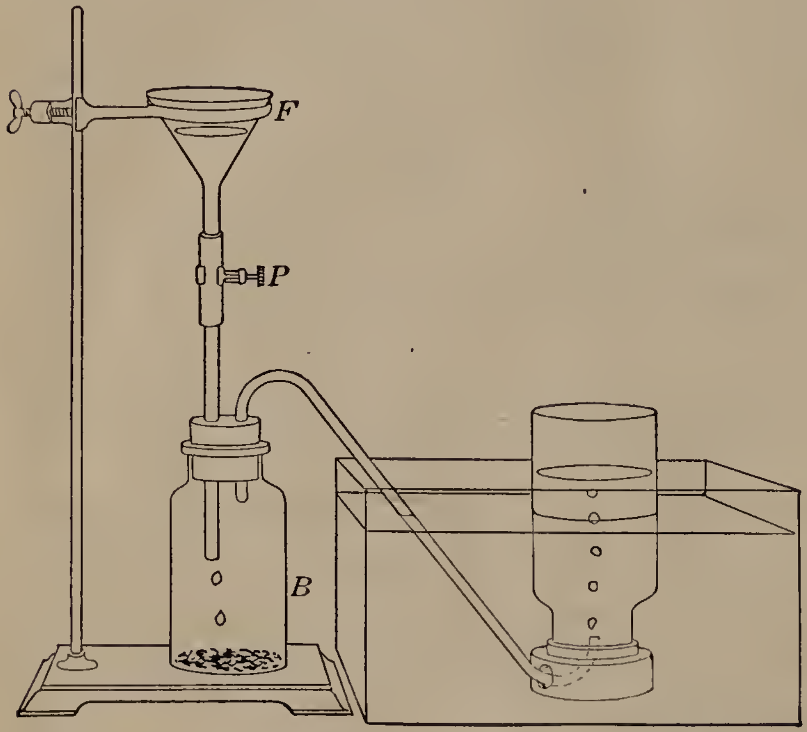


Fig. 21. Making oxygen from sodium peroxide and water.

EXPERIMENT 6

Percentage of Oxygen in Potassium Chlorate

Apparatus. — Porcelain crucible and cover. Ring stand. Horn pan balance. Set of weights. Pipe-stem triangle. Iron forceps. Bunsen burner.
Materials. — Dry potassium chlorate.

Clean and dry a porcelain crucible and cover. Weigh the crucible empty to a centigram (0.01 g.). Put about 2 grams of *dry* powdered potassium chlorate in the crucible and weigh again as before.

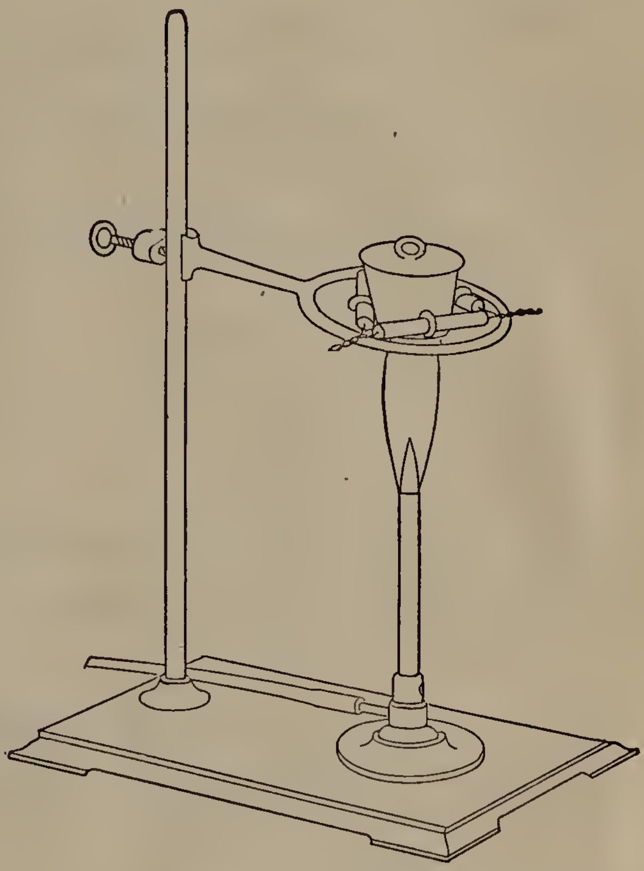


Fig. 22. **Heating potassium chlorate in a crucible.**

Place the covered crucible on the pipe-stem triangle and support it on the ring stand at some convenient height (Fig. 22). Begin heating with a small Bunsen flame (about 5 cm. high) very gently. Lift the cover occasionally with the forceps and adjust the flame so that the melted potassium chlorate gives off its oxygen slowly. In case some should spatter up on the cover, withdraw the flame and remove the cover to a clean piece of smooth paper. Loosen the bits of solid matter with a pin and very carefully return them to the crucible. Continue heating until the bubbling slackens, then heat strongly for 10 minutes. Now lower the flame to permit the crucible to cool gradually and finally remove it. When cold, weigh to the nearest centigram (0.01 g.).

Again heat very hot for about 5 minutes, cool, and weigh as before. If the weight has not changed during the second heating, we may consider that the oxygen has been completely expelled. If the weight is less, heat once again for 5 minutes and continue in this way until two successive weights are the same. This process is called **heating to constant weight.**

The loss of weight is equal to that of the oxygen. The residue left in the crucible is called **potassium chloride.**

The percentage of oxygen in potassium chlorate may be calculated thus:

Wt. of potassium chlorate : wt. of oxygen : : 100 : x

Make all computations in your notebook. Arrange your work in tabular form:

Wt. of crucible + potassium chlorate . . . . g.
Wt. of crucible empty . . . . g.
  Wt. of potassium chlorate . . . . g.
Wt. after first heating . . . . g.
Wt. after last heating . . . . g.
  Wt. of oxygen . . . . g.

Find the average of the percentages found by all in the class who have done this experiment. *What law of chemistry is thus illustrated?*

Optional work. The same method may be used to obtain the percentage of oxygen in sodium chlorate.

EXPERIMENT 7

Decomposition of Water by Sodium

Apparatus. — Knife. Evaporating dish. Forceps. Ring stand and clamp. Pneumatic trough. Large test tube. Glass plate. Stirring rod.

Materials. — Sodium. Litmus paper strips, red and blue. Lead foil.

Caution. The reaction of sodium with water is violent. Don't handle sodium with wet hands. Follow the directions.

a. What is the appearance of a freshly cut surface of sodium? *Is sodium hard or soft? heavy or light?*

b. Fill an evaporating dish one-fourth full of water, and drop into it with the forceps a bit of sodium (size of the head of a match). It is well to protect the eyes from spattering, with a plate of glass (Fig. 23). *Result?*

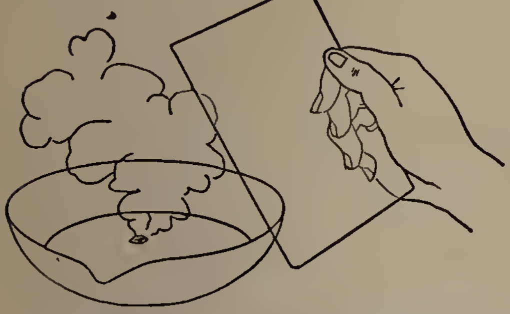


Fig. 23. Violent action of sodium on water.

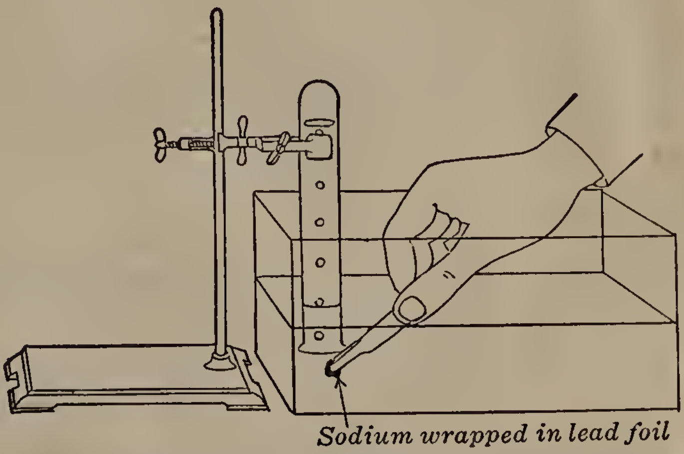


Fig. 24. Collecting the gas evolved from sodium and water.

Throw in another similar piece of sodium. Apply a lighted match, held at arm's length, to the sodium while it is reacting on the water. *Result?*

c. After the action has ceased, wet your fingers with the solution and rub them together. *Result?* Test with a stirring rod the action of a drop of the solution on red and blue litmus paper. *Result?*

d. Wrap a clean, freshly cut piece of sodium (about 3 mm. in diameter) in dry lead foil (tea lead). Punch several holes in the lead with a knife blade. Holding the lead in forceps, quickly slip it under the mouth of an inverted large test tube filled with water (Fig. 24). If necessary, use another piece of sodium wrapped in lead to complete the filling of the tube. If the sodium escapes from the lead covering, stand aside.

When the tube is filled with gas, carry it mouth downward to a flame and ignite the gas. *Result?* This gas is hydrogen, and the yellow flame is due to sodium. *If sodium is an element, where does the gas come from?*

Draw a diagram of the apparatus used in **d.**

Optional work. Calcium is also a metal which liberates hydrogen from water. The reaction is not so violent as that of sodium with water. Hence it is perfectly safe and furnishes a better experiment than sodium. Calcium does not have to be wrapped in lead foil and can be handled much as other common metals.

NOTE. Metallic calcium may be cut up into small pieces by fixing it in a vise and chipping it off with a chisel.

EXPERIMENT 8

HYDROGEN — PREPARATION AND PROPERTIES

Apparatus. — Wide-mouth bottle with 2-hole stopper. Thistle tube. Pneumatic trough. Glass tubing. 3 wide-mouth bottles and glass squares. Bunsen burner. Test tube.

Materials. — Zinc. Dilute sulfuric acid. Copper sulfate solution. Wax tapers.

I. Preparation. A convenient method of preparing hydrogen is furnished by the action of zinc on dilute sulfuric acid. Place about 20 g. of zinc in a bottle provided with a two-hole rubber stopper carrying a thistle tube and a delivery tube. The apparatus is arranged for collecting the gas over water (Fig. 25).

Insert the stopper tightly with a twisting motion and pour through the thistle tube enough dilute sulfuric acid to cover the zinc. **Be sure that the thistle tube dips into the acid.** If the gas does not come off freely, add through the thistle tube a few drops of copper sulfate solution.

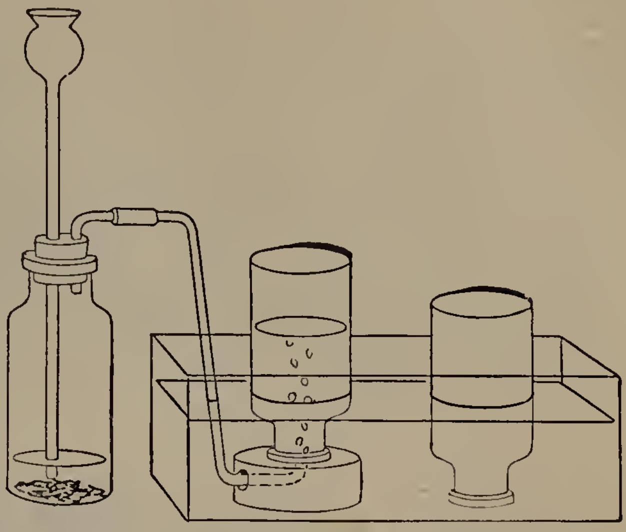


Fig. 25. Generating hydrogen by the action of zinc on dilute sulfuric acid.

The gas which passes over first is evidently a mixture of hydrogen and air. A confined mixture of hydrogen and air, if ignited, explodes with great violence. To determine when the hydrogen is free from air, collect a test tube full of the gas and carry it mouth downward to a small flame and ignite the gas. Continue to collect and test the gas in this way until a portion does not give a sharp explosion but burns quietly. Then collect 3 bottles of hydrogen, cover each with a glass plate, and set mouth downward on the desk.

II. Properties. *a.* Allow one bottle of hydrogen to stand uncovered (mouth up) for a minute. Then bring a lighted taper to the mouth of the bottle. *What does this show about the density of hydrogen as compared with air?*

b. Light a short taper and lift a second bottle of hydrogen (mouth downward). Insert the burning taper almost to the bottom of the bottle and then withdraw it slowly. *What happens to the flame of the taper? What occurs at the mouth of the bottle? Explain.*

c. Place a bottle of hydrogen, mouth downward, over a bottle of air, mouth upward. Allow the bottles to remain mouth to mouth in this position for 3 minutes. Carry first the lower bottle and then the upper bottle quickly to a flame. *What fact about the diffusion of gases does this experiment illustrate?*

d. Summarize *the properties of hydrogen and compare with those of oxygen.*

Optional work. Filter the liquid remaining in the flask (the black specks in the liquid of the generator are impurities, mainly carbon, which were in the zinc), and evaporate to about one-half its volume in a porcelain dish over asbestos. Cool and allow the liquid to stand until crystals form. They are called **zinc sulfate** and contain zinc, sulfur and oxygen. *If sulfuric acid contains hydrogen, sulfur, and oxygen, how would you describe the chemical change that takes place in the gas-generating bottle?*

EXPERIMENT 9

REDUCTION OF AN OXIDE WITH HYDROGEN

Apparatus. — Wide-mouth bottle with 2-hole stopper. Thistle tube. Drying tube with stopper. Ring stand and clamp. Bunsen burner. Test tube. Glass tubing.

Materials. — Copper oxide (wire form). Zinc. Dilute sulfuric acid. Absorbent cotton. Fused calcium chloride.

a. We have already seen that bright metallic copper turns to black copper oxide when heated in the air (Exp. 1). In this experiment we shall reverse the process of oxidation and change copper oxide back to metallic copper; this is called **reduction.**

For this purpose we shall use dry hydrogen (free from water vapor) which can be obtained by passing the gas from the generator through a tube filled with granulated calcium chloride. This drying material is held in place by loose plugs of cotton placed at each end of the tube.

b. Place about 0.5 cc. of black copper oxide in the bottom of a dry test tube and place the latter in a slightly inclined position with mouth downward (Fig. 26). Insert a straight piece of glass tubing into the stopper of the drying tube and introduce this tubing into the test tube until the end touches the copper oxide.

c. Now generate hydrogen as in Experiment 8, and wait 3 minutes to allow all the air to be replaced by hydrogen.

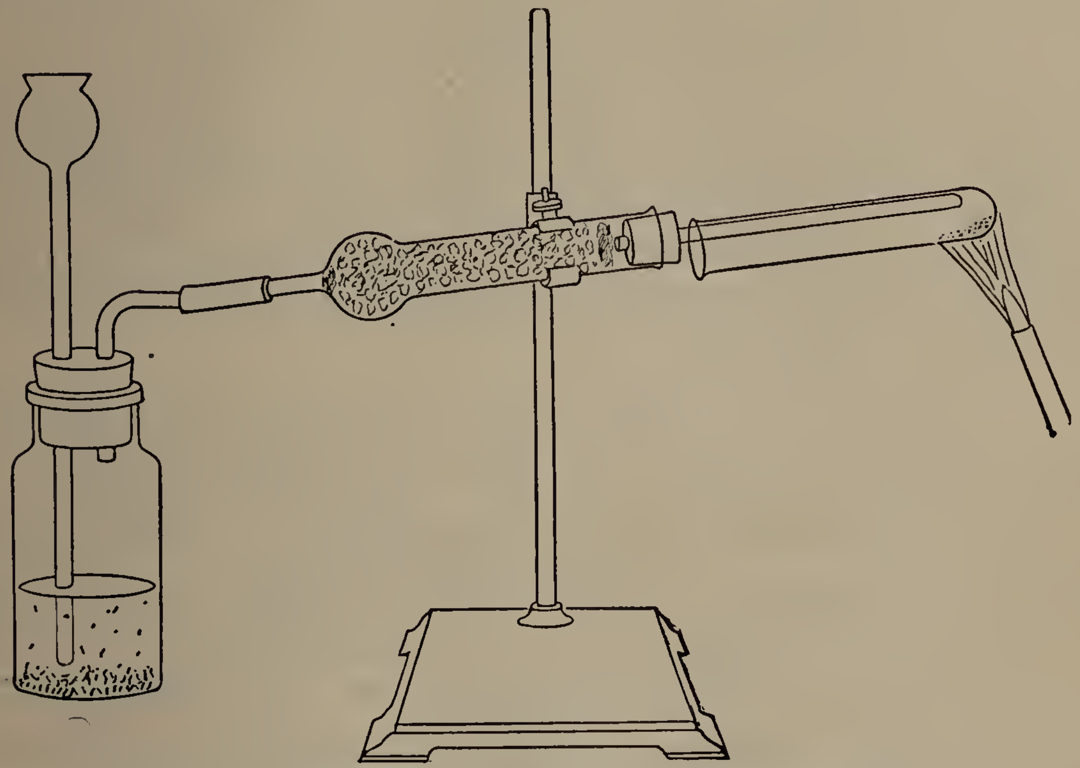


Fig. 26. Reducing copper oxide with dry hydrogen.

Caution. Have no flame near the apparatus until all the air is driven out.

Cautiously heat the copper oxide to redness, being careful to keep the flame away from the mouth of the tube.

Note any change in the copper oxide. What collects in the cool part of the tube? What substance is reduced and what substance is oxidized in this experiment?

Why is it necessary to dry the hydrogen?

Optional work. Try this experiment using ferric oxide instead of copper oxide.

EXPERIMENT 10

WEIGHT OF A LITER OF OXYGEN

Apparatus. — Iron crucible. Ring stand and clamp. Pneumatic trough. 2-liter bottle (acid bottle). Test tube with 1-hole stopper. Horn pan balance and weights. Bunsen burner. Glass plate. Graduated cylinder. Glass tubing.

Materials. — Manganese dioxide (powdered). Well dried potassium chlorate. Glass wool.

a. In this experiment we first determine the weight of a definite volume of oxygen and then compute the weight of one liter measured under standard conditions. To determine the weight of the oxygen, we weigh the generating apparatus both before and after a definite volume of oxygen has been produced. To measure the volume of the oxygen, we measure the volume of water which it displaces and then, knowing the temperature and barometric pressure, we can compute according to the Gas Laws [§ **47**] * the volume under standard conditions (0° C. and 760 mm.).

b. It is important in this experiment to make sure that the loss of weight in the generating apparatus is due to the oxygen evolved and not in part to moisture driven off from the materials used. Therefore it is well to heat about 6 g. of pulverized manganese dioxide in an iron crucible (uncovered) for 5 minutes, stirring it occasionally.

In the meantime, fill an acid bottle (about 2 liters capacity) with water and invert it in the pneumatic trough. Insert a delivery tube in a one-hole rubber stopper which fits a dry test tube.

When the manganese dioxide has cooled, mix it on a sheet of paper with 7 g. of dry pulverized potassium chlorate. Slide the mixture into the test tube and place a plug of glass wool or

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* Bracketed numbers refer to sections in *Black and Conant's* Practical Chemistry.

well ignited asbestos fiber in the mouth of the tube to catch small particles carried along by the gas. Weigh the test tube with its contents to the nearest centigram (0.01 g.) and record the result according to the following table. Replace the stopper in the test tube and test for air-tightness as follows: place the end of the delivery tube in the mouth and suck out some of the air. If the tip of the tongue seems to adhere to the tube, the apparatus is tight. Arrange the apparatus as shown in figure 27.

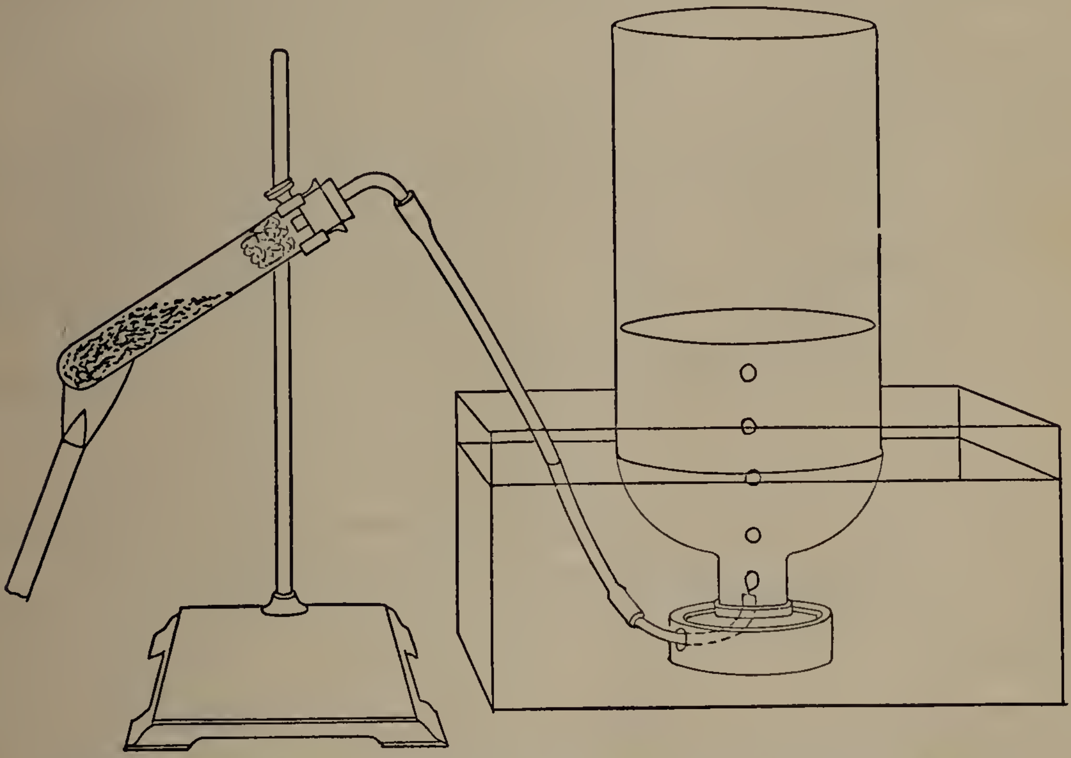

Fig. 27. Apparatus for finding the weight of a liter of oxygen.

c. Heat the contents of the test tube with a small flame, beginning at the top and gradually working downward. Collect all the gas evolved in the bottle. **Don't rush! Regulate the heating so that you could count the bubbles of oxygen.**

When the bottle is nearly full, or when the oxygen has stopped coming over, remove the delivery tube from the water and allow the test tube to cool.

d. In the meantime, to measure the volume of the oxygen, first equalize the level of the water inside and outside the bottle. To do this, it may be necessary to lower the bottle and incline it. Then, while the bottle is in this position, close it with a stopper or glass plate and set it upright on the table.

To measure the volume of gas collected in the bottle, measure the amount of water needed to fill the bottle, pouring the water into the bottle from a graduated cylinder. Record the total volume of water used, the temperature of the water in the pneumatic trough, and the barometric pressure.

When the test tube has cooled so that it feels barely warm, weight the test tube and its contents as before and record the weight. Compute the loss of weight, which is the weight of oxygen evolved.

Correct the barometric pressure for aqueous tension [§ **48**] and then reduce the observed volume of oxygen from the observed temperature and pressure to 0° C. and 760 mm.

From this reduced volume and the weight, compute the weight of 1 liter of oxygen (density).

Table

Wt. of test tube + contents before heating	g.
Wt. of test tube + contents after heating	g.
Wt. of oxygen evolved	g.
Temperature	°C.
Volume of oxygen under conditions of exp.	cc.
Barometric pressure	mm.
Pressure due to water vapor	mm.
Corrected barometric pressure	mm.
Volume of oxygen under stand. conditions	cc.
Density of oxygen (wt. of 1 liter)	g.

EXPERIMENT 11

DISTILLATION OF WATER

Apparatus. — Flask with 1-hole stopper. Ring stand. Wire gauze with asbestos center. Bunsen burner. Wide-mouth bottle. Test tubes. Glass tubing.

Materials. — Common salt (sodium chloride). Potassium permanganate. Ammonia water (concentrated). Litmus paper (red). Marble chips or glass beads.

a. Dissolve a spoonful of common salt in about 50 cc. of water. Put the solution in a flask which is arranged as shown in figure 28. A few chips of marble in the flask will prevent "bumping." The steam from the boiling water in the flask is condensed by conducting it into the test tube which is kept cold by the water in the bottle. Boil gently until 5 cc. has distilled over. Taste the distilled water. *Is it free from salt? Explain.*

Repeat using 50 cc. of water colored with a crystal of potassium permanganate. *Is the distillate colorless?* (Don't taste it.)

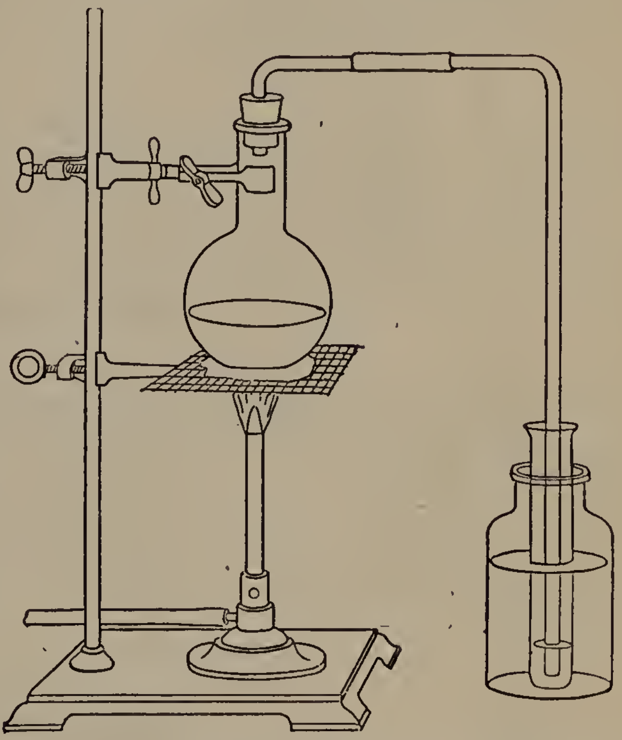

Fig. 28. Simple apparatus for distilling water.

b. Add a few drops of concentrated solution of ammonia (a gas) to 50 cc. of water. Note the odor of the liquid and place a drop of it on red litmus paper. *Result?*

Distill the dilute ammonia water in the same way in which you did the salt water. *What is the odor of the distilled liquid?* Test it with red litmus paper. *Is it free from ammonia? Explain.*

Optional work. Set up a Liebig condenser as represented in figure 29. Use 100 cc. of ordinary water and add to it one drop of concentrated ammonium hydroxide solution. In each of six clean test tubes place one drop of phenolphthalein solution (an indicator which turns pink with ammonia) and catch the distillate in these test tubes. Change the test tube as soon as 15 cc. of liquid has come over. *Does the ammonia pass over with the steam? Do the first portions of the distillate contain more or less ammonia than the later portions?*

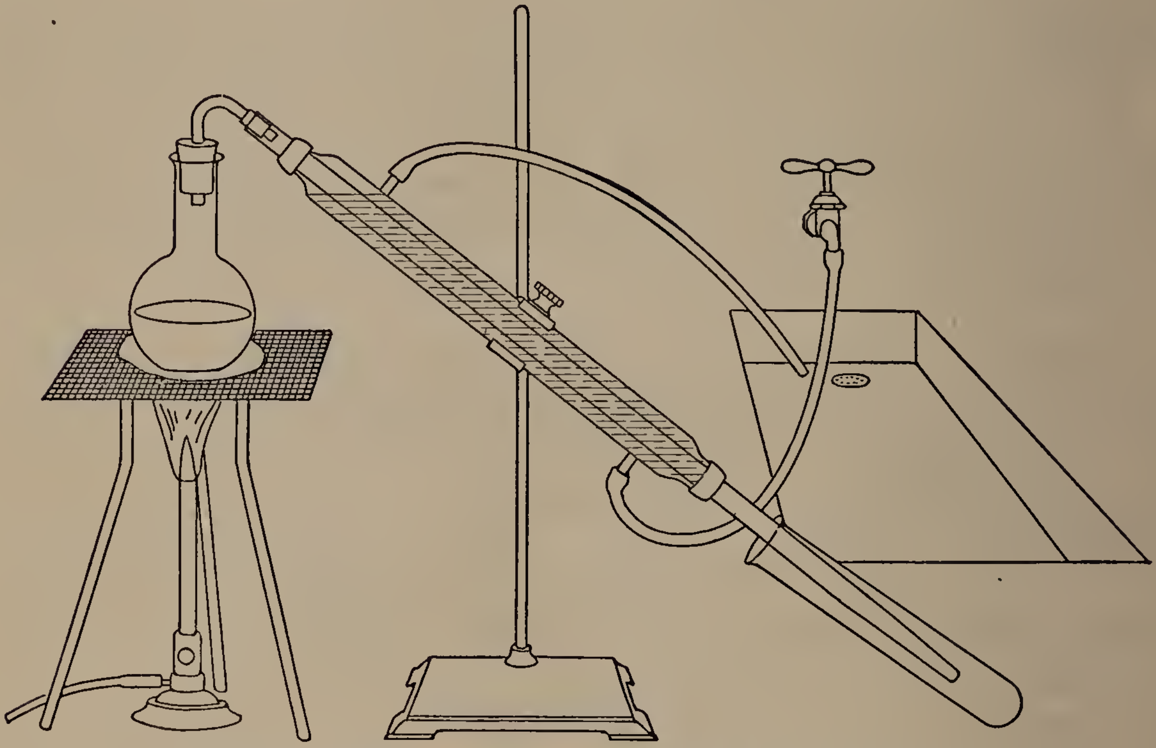

Fig. 29. **Distilling water with Liebig condenser.**

Calcium Carbonate

EXPERIMENT 12

CARBON DIOXIDE — PREPARATION AND PROPERTIES

Apparatus. — Wide-mouth bottle with 2-hole stopper. Thistle tube. 3 wide-mouth bottles and glass plates. Glass and rubber tubing.
Materials. — Marble chips. Dilute hydrochloric acid. Litmus paper (blue). Tapers. Limewater. Splint.

a. Preparation. Use the same apparatus which you used to generate hydrogen, and arrange to collect 3 bottles of gas by displacement of air (Fig. 30). Slide into the bottle (don't drop them in) enough marble chips to cover the bottom. Add dilute hydrochloric acid through the thistle tube until a brisk evolution of gas is obtained. To test when a bottle is filled, bring a burning splint to its mouth; the gas will extinguish the flame. Cover each bottle with a glass plate. Allow the gas to bubble through a little water in a test tube. Taste the liquid and test it with litmus paper.

Fig. 30. Generating carbon dioxide and displacing the air.

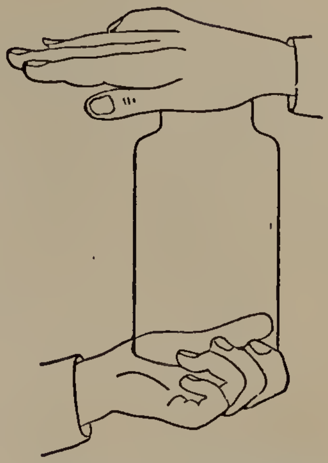

Fig. 31. Shaking carbon dioxide with water.

b. Properties. Light a taper and pour carbon dioxide from one of the bottles over it as you would pour water. *Does the gas support combustion? Is the gas heavier or lighter than air?*

c. Pour about 10 cc. of water into a bottle of the gas, cover at once tightly with the palm of the hand (Fig. 31), and shake it vigorously. *Is the hand sucked in, that is, held against the mouth of the bottle by atmospheric pressure? Is the gas soluble?*

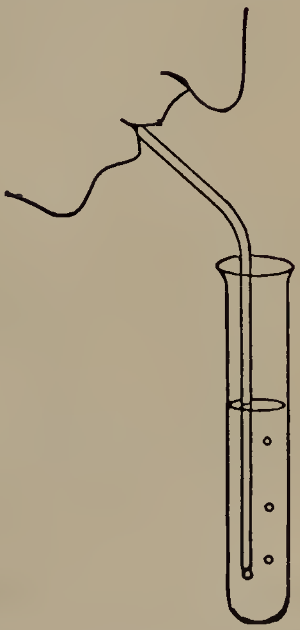

Fig. 32. Blowing through limewater.

d. Pour about half a test tube full of clear limewater into a bottle of carbon dioxide and shake the bottle. The milkiness is due to the calcium carbonate which is precipitated. This serves as a good **test for carbon dioxide.**

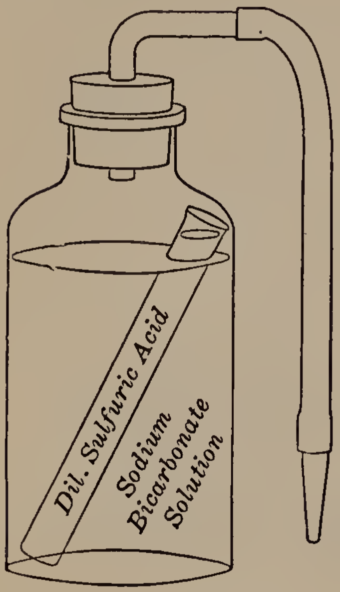

Fig. 33. Simple form of chemical fire extinguisher.

e. Burn a pine splint in a bottle of air. Add a little limewater and shake the bottle. *What gas is formed when wood burns?*

Blow through a glass tube into a test tube half full of limewater (Fig. 32). *What gas does the air exhaled from the lungs contain?*

f. *Make a list of the properties of carbon dioxide which you have observed.*

Optional work. The construction and operation of one form of fire extinguisher may be shown by the apparatus in figure 33. The bottle is nearly filled with a saturated solution of sodium bicarbonate (baking soda), and the small test tube with very dilute sulfuric acid (about 1 to 10).

To show its operation, build a little fire of paper and splints in a pan and then grasp the bottle so as to hold the stopper in firmly and invert. Put out the fire. *Explain its operation.*

EXPERIMENT 13

Carbon Monoxide — Preparation and Properties

Apparatus. — Ring stand with 1 ring and clamp. Wire gauze with asbestos center. Bunsen burner. Small flask with 2-hole stopper fitted with delivery tube and dropping funnel. Pneumatic trough. 3 wide-mouth bottles and glass squares.

Materials. — Concentrated sulfuric acid. Formic acid. Taper. Limewater.

Caution. **Carbon monoxide is poisonous. Do not inhale it.**

a. Preparation. Fit a small flask with a two-hole stopper carrying a delivery tube and a funnel connected by a short rubber tube and pinchcock with a straight tube as shown in figure 34. Set up the apparatus on a ring stand with the asbestos wire netting under the flask at such a height as to heat the flask very gently with a small flame. Arrange the delivery tube for collecting the gas over water. Remove the stopper from the flask and pour in 15 cc. of concentrated sulfuric acid. Replace the stopper and partially fill the funnel with formic acid. Never let this run out entirely during the experiment.

Allow a few drops of the formic acid to flow into the flask and warm very gently. Adjust the pinchcock so as to admit the formic acid one drop at a time and so as to get a steady flow of gas from the flask. Collect 3 bottles full of the gas and close the pinchcock to stop the generation of gas.

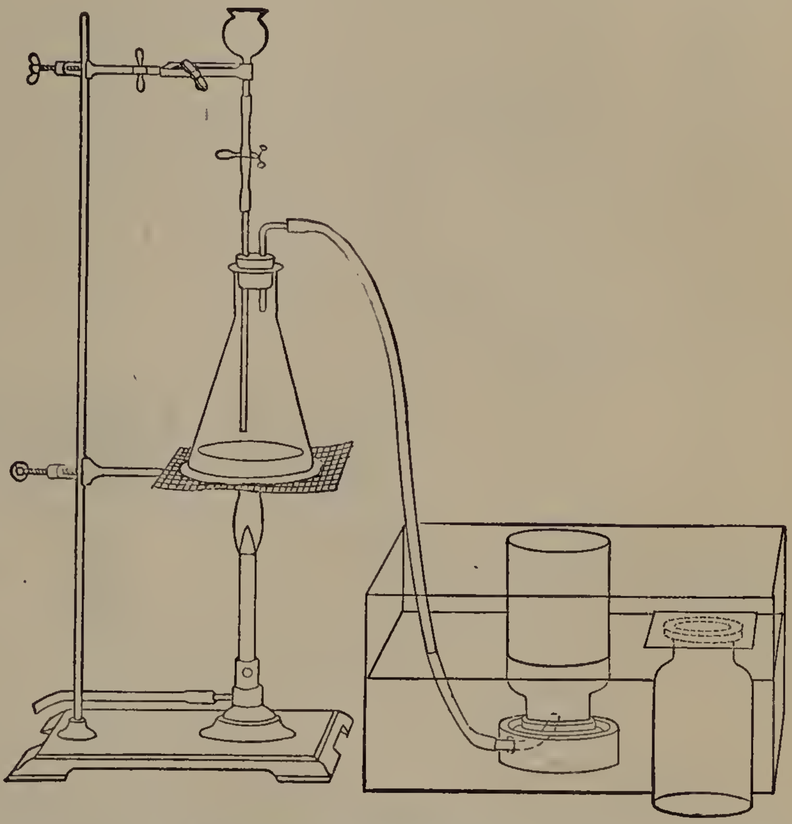

Fig. 34. Generating carbon monoxide by dropping formic acid on warm concentrated sulfuric acid.

b. Properties. Do not use the first bottle. *Why not?* Light a short taper and lift one bottle up mouth downward. Plunge the burning taper up into it. *Does the gas burn? Does the taper burn in the gas?*

c. Take another bottle of the gas and slip the glass plate partly off in order to pour into the bottle 5 cc. of clear limewater; then quickly replace the plate and, holding it tightly against the mouth of the bottle, shake the contents of the bottle. *Note any change in the limewater.* Now light a taper, remove the glass plate, and quickly ignite the gas. Replace the cover at once and when the flame dies out, shake the contents again. *What gas is formed when carbon monoxide burns?*

d. Summarize *the properties of carbon monoxide. How could you distinguish burning carbon monoxide from burning hydrogen?*

Fig. 35. Reducing copper oxide with carbon monoxide.

Optional work. The reducing action of carbon monoxide may be shown by passing the gas through water and then over hot copper oxide (Fig. 35). The carbon dioxide which is formed is shown by the milkiness produced in limewater. Any unchanged carbon monoxide is collected over water and then burned.

EXPERIMENT 14

ATMOSPHERIC NITROGEN

Apparatus. — A 2-liter bottle with 2-hole stopper. Hard-glass tubing (20 cm.) with two 1-hole stoppers. Bunsen burner. 3 wide-mouth bottles. Pneumatic trough. Ring stand and clamp. Tubing.

Materials. — Copper gauze or turnings.

a. Preparation. Since the oxygen of the air combines readily with hot copper and the nitrogen does not, it is easy to obtain nitrogen from air. Set up the apparatus as shown in figure 36.

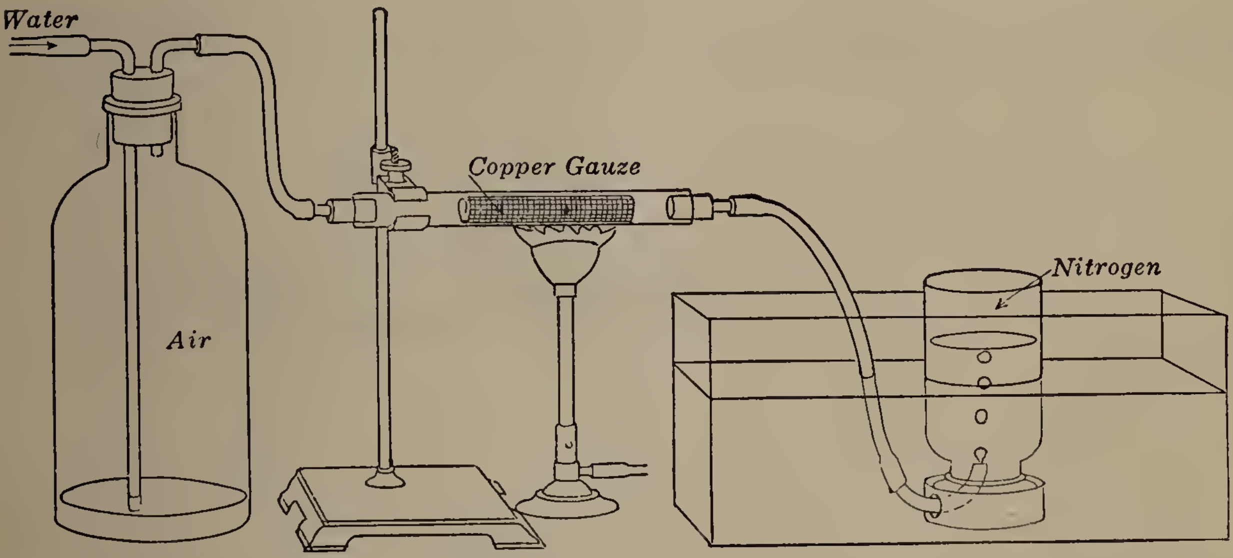

Fig. 36. Passing air over hot copper.

The hard-glass tube contains copper gauze or clean copper turnings. The current of air is obtained by letting water from the tap fill the bottle.

b. Heat the copper at first gently by moving the flame along the tube and then strongly. Do not burn the stopper. Carefully turn on the water so as to drive the air from the bottle very slowly over the hot copper. Collect three bottles of gas.

c. Properties. Apply a lighted taper to a bottle of the gas collected. *What happens?* Leave the bottle uncovered for 3 minutes and test again. *Does the taper now burn in the gas or not? Conclusions?*

Add a little clear limewater to another bottle and shake the contents. *Does the limewater turn milky?*

Find out whether burning sulfur will continue to burn in nitrogen.

Make a list of the properties of nitrogen which you have observed.

What is the black substance seen on the copper after cooling?

How can you distinguish nitrogen from carbon dioxide?

Why was the air passed over the copper ***slowly****?*

What impurity does the nitrogen thus prepared contain? [§ **81**]

Optional work. By using copper and magnesium in a longer hard-glass tube (30 cm. long), it is possible to absorb both the oxygen and the nitrogen of air. Put a loose plug of shredded asbestos in the middle of the tube with copper gauze on the left side and magnesium powder on the right. Heat both metals as shown in figure 37 and then very slowly pass a steady stream of air through the tube from left to right. Continue as long as there is evidence of a change in both metals. Break the tube, *examine and name the products.* Place the magnesium product in water. *Note any odor.*

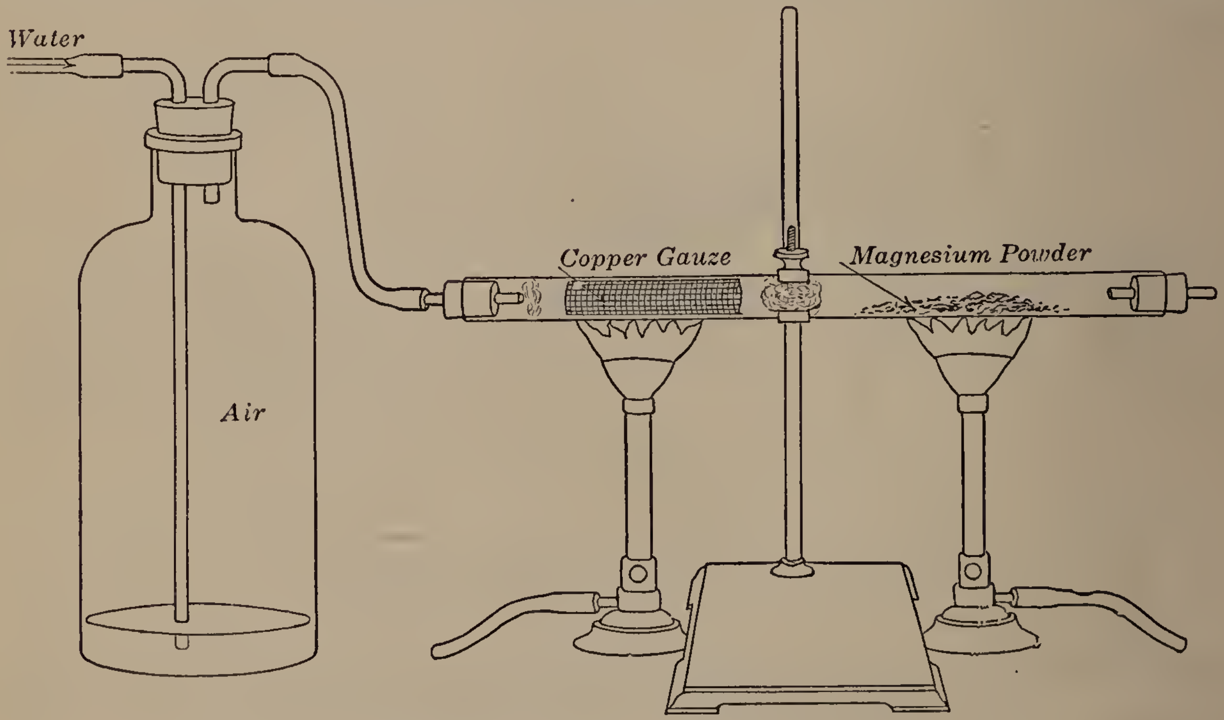

Fig. 37. **Passing air over hot copper and magnesium.**

EXPERIMENT 15

PERCENTAGE OF OXYGEN IN AIR — VOLUMETRIC

Apparatus. — Pneumatic trough. Ring stand with clamp. 100 cc.-graduated cylinder. Thermometer. Barometer.
Materials. — White phosphorus. Copper wire (30 cm. # 18).

a. A convenient method of determining what percentage of the air is oxygen consists in burning some substance in a definite volume of air and then measuring the volume of the remaining gas. White phosphorus is a good substance for this purpose because it forms an oxide which is soluble in water.

CAUTION. Great care must be taken never to handle white phosphorus except under water.

b. To get a definite volume of air invert a 100 cc.-graduated cylinder so that its mouth dips under the surface of the water in a pneumatic trough. The cylinder may be tipped to let part of the inclosed air escape, and then it should be securely clamped in a vertical position so that the water level inside and outside the cylinder is the same. *The volume of the air thus confined* (about 100 cc.) *should be carefully observed and recorded.* Also *record the barometric pressure and the temperature of the water,* which should have stood long enough to be at the temperature of the room.

c. A piece of ordinary (white) phosphorus should now be stuck on the end of a piece of copper wire (under water) and the wire carrying the phosphorus should be pushed up into the air space in the cylinder and allowed to stand overnight. It is well to arrange the apparatus (Fig. 38) so that the phosphorus does not touch the sides of the tube and stands well up toward the top of the cylinder.

Applying the Gas Laws, compute the volume that this air would occupy under standard conditions (0° C. and 760 mm.).

d. The next day remove the phosphorus from the cylinder and lower the cylinder until the level of the water inside is the same as that outside. Read and *record the volume of the gas remaining in the cylinder,* also *the temperature of the water and the barometric pressure.*

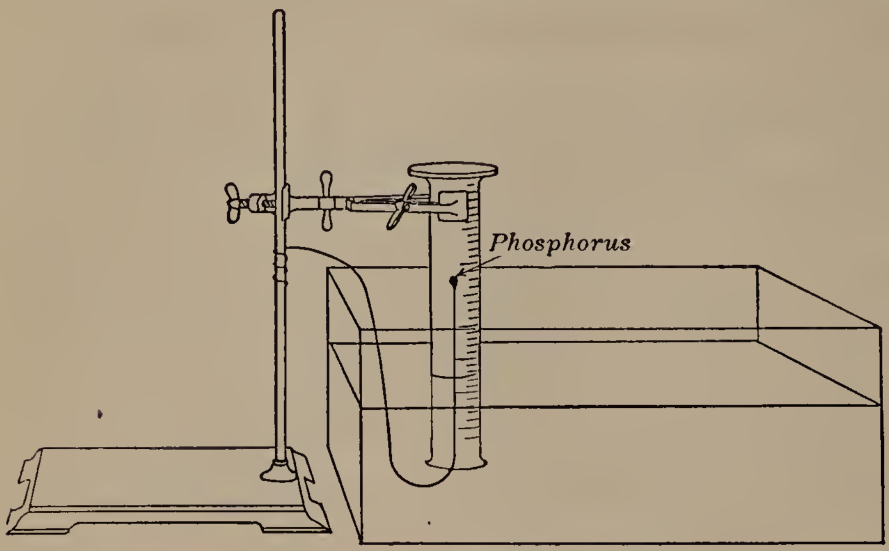

Fig. 38. Burning the oxygen out of air with phosphorus.

e. Compute the volume which the remaining gas would occupy if it were under standard conditions. Then calculate the difference between the original volume of air inclosed and the volume of gas that remains (both at standard conditions). This represents *the volume of oxygen* removed by the phosphorus.

Calculate the percentage of oxygen in air as follows:

Volume of air : Volume of oxygen : : 100 : x

f. *What properties did you observe in the gases left after the oxygen had been absorbed? What were the gases?*

Make all calculations in your notebook and record your observations and the results of your calculations in tabular form:

Volume of air taken	cc.
Temperature of the water, first day	°C.
Barometric pressure, first day	mm.
Volume of air at 0° C. and 760 mm.	cc.
Volume of gas remaining in cylinder	cc.
Temperature of water, second day	°C.
Barometric pressure, second day	mm.
Volume of gas remaining at 0° C. and 760 mm.	cc.
Volume of oxygen absorbed by phosphorus	cc.
Percentage of oxygen in air (by volume)	%

Optional work. Another quicker method of absorbing the oxygen from air is by means of an alkaline solution of pyrogallol. This method. however, requires considerable dexterity to get good results.

Set up the apparatus as in figure 39. Disconnect the large test tube temporarily from the stopper. Mix 3 cc. of pyrogallic acid solution with 20 cc. of sodium hydroxide solution and pour this into the funnel. Now open the pinchcock slightly and permit the solution to fill the rubber and glass tubes completely down to the opening of the nozzle. Replace the test tube, fitting the stopper tightly into its mouth, and thus inclose a volume of air equal to the capacity of the test tube.

Now open the pinchcock. A little of the solution will be forced into the test tube by the pressure of the liquid above, and, as the oxygen is absorbed, more will enter. When the liquid stops flowing in, close the pinchcock and turn the test tube upside down several times to absorb the last traces of oxygen. Care must be taken not to warm the tube by handling.

Finally while the test tube is in the inverted position, reopen the pinchcock and equalize the levels of the liquid in test tube and funnel by raising or lowering the former. Then close the pinchcock, restore the test tube to its original position, and mark the positions of the bottom of the stopper and of the surface of the liquid by means of rubber bands.

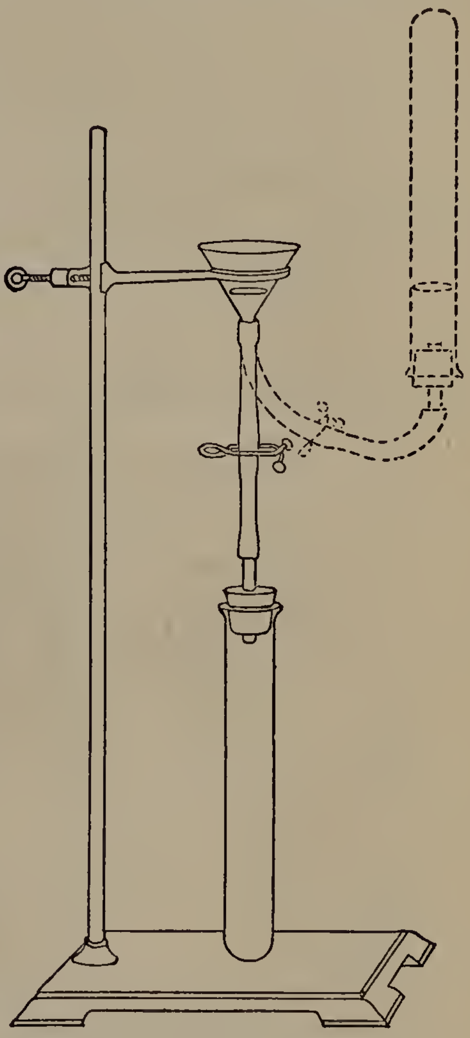
Fig. 39. Absorption of oxygen in the air by pyrogallol.

Clean the apparatus and measure with a graduated cylinder the volume of the test tube up to the lower and upper bands. The former is the volume of the oxygen, and the latter that of the air. Calculate *the percentage of oxygen in air* (by volume), *i.e.* the number of cubic centimeters of oxygen in 100 cubic centimeters of air.

EXPERIMENT 16

REACTING OR EQUIVALENT WEIGHT OF A METAL

To find the weight of zinc needed to generate 1 g. of hydrogen

Apparatus. — Small flask with 1-hole stopper. Pneumatic trough. Liter bottle. Glass square. Graduated cylinder. Thermometer. Barometer.

Materials. — Concentrated hydrochloric acid. Pure sheet zinc. Sand paper.

a. To determine the reacting weight (sometimes called equivalent weight) of a metal, we need (1) to take a known weight of the metal, (2) to displace hydrogen from an acid with this quantity of the metal, (3) to measure the volume of hydrogen displaced, and (4) knowing the density of hydrogen (grams per liter), to calculate the weight of this hydrogen.

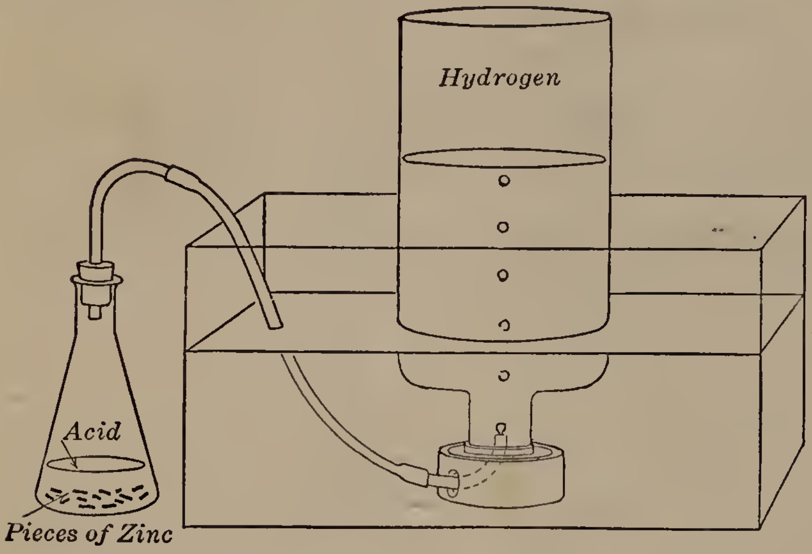

Fig. 40. Finding the reacting weight of a metal by replacing hydrogen in an acid.

b. Set up the apparatus as shown in figure 40, using a small flask which is provided with a tightly fitting one-hole rubber stopper and exit tube leading to the pneumatic trough. Fill the liter bottle with water and invert it in the pneumatic trough so that the water may be at room temperature. Place in the flask 10 cc. of concentrated hydrochloric acid and 20 cc. of water. Clean scrupulously with sand paper about 2 or 2.5 grams of the purest sheet zinc, wipe it clean, and weigh to a centigram (0.01 g.).

c. Drop the zinc into the acid and introduce the stopper as quickly as possible. Collect all the gas evolved. When the action has ceased, raise or lower the bottle in the pneumatic trough until the water level is the same inside and outside the bottle. To do this it may be necessary to incline the bottle. While it is in this position, close it with a stopper or glass plate and set it right side up on the table. Measure the volume of gas by pouring in water from a graduated cylinder.

d. Record the temperature of the water and the barometric pressure; then apply the Gas Equation [§ 47] and compute the volume of hydrogen evolved under standard conditions (0° C. and 760 mm.).

e. Assuming that *the weight of one liter of hydrogen under standard conditions is 0.09 grams,* calculate the weight of the hydrogen given off by the zinc. Finally, calculate from the weight of zinc used and the weight of hydrogen evolved, *the weight of zinc needed to give one gram of hydrogen;* thus:

Wt. of zinc used : wt. of hydrogen evolved : : x : 1

Make all calculations in your notebook and tabulate your data and results as follows:

Weight of zinc taken . . . . g.
Volume of hydrogen obtained . . . . cc.
Temperature of water . . . . °C.
Barometric height . . . . mm.
Pressure due to water vapor (p. 157) . . . mm.
Pressure of hydrogen, corrected . . . . mm.
Volume of hydrogen at 0° C. and 760 mm. . . . . cc.
Weight of this hydrogen . . . . g.
Weight of zinc needed to get 1 g. of hydrogen . . . . . g.

Optional work. In the same way it is possible to determine the reacting weight of other metals, such as *aluminum* (use about 0.7 g.), *iron* (use piano wire, about 0.9 g.), or *magnesium* (about 0.9 g.) Since magnesium ribbon is very nearly uniform in size, its weight may be considered proportional to its length. It is well to measure off 5 meters of clean, bright ribbon, to weigh it carefully, and from this data compute the weight of 1 cm. Then the required weight of magnesium can be measured off. In case a liter-bottle is not at hand, use an ordinary 250 cc. wide-mouthed bottle and 0.18 g. of magnesium. It will also be interesting to use *dilute sulfuric acid* (adding 10 cc. concentrated acid to 30 cc. water) and compare results.

If a 100 cc. measuring tube and battery jar are at hand, this experiment can be quickly done as follows: Calculate the length of magnesium required to give 0.08 g. Pour 15 cc. of concentrated hydrochloric acid into the measuring tube and then fill the tube with water, taking care not to mix the water and acid more than is necessary. Coil the magnesium ribbon into a small space, place it in the tube, cover it quickly with the thumb, invert, and open under water in a small battery jar. If any metal sticks to the sides of the glass as the liquid goes down, rinse it off by shaking the glass. When the action is complete, equalize the water levels and read directly the volume of hydrogen.

EXPERIMENT 17

HYDROCHLORIC ACID

Apparatus. — Ring stand with clamp. Bunsen burner. Asbestos gauze. Florence flask with 2-hole stopper. Thistle tube. Wide-mouth bottles and glass squares. Glass tubing. Cardboard. Dish. Stirring rod.

Materials. — Sodium chloride (common salt). Concentrated sulfuric acid. Litmus papers. Concentrated ammonium hydroxide. Splint. Filter paper. Magnesium.

a. Preparation. Arrange a flask with stopper, thistle tube, and delivery tube as shown in figure 41. Pour into the flask 7 cc. of water and then pour in **slowly** 20 cc. of concentrated sulfuric acid (H_2SO_4).

CAUTION. **Always pour the sulfuric acid into the water.**

Cool the diluted acid by holding the flask in a stream of running water. When the acid is cold put into it about 15 g. of common salt (NaCl). Replace the stopper carrying the thistle tube and delivery tube and set the flask on the asbestos gauze. Clamp the neck of the flask at the right height for heating with a Bunsen burner. The thistle tube must extend almost to the bottom of the flask so that its end may be under the liquid. Put the delivery tube through a piece of cardboard and let it extend nearly to the bottom of a dry clean bottle.

Heat the flask very slowly with a small flame. You can tell when the bottle is full of the hydrogen chloride gas by holding a strip of moist blue litmus paper at the mouth of the bottle. When the paper turns red, the bottle is full of the gas (HCl) and should be replaced at once by another. Collect three bottles of gas and cover them with glass plates. Then fill another bottle about one-third full of distilled water and place the end of the delivery tube just below the surface of the water. Continue heating and observe the currents in the water at the end of the delivery tube. *What causes them?* Avoid letting the hydrogen chloride gas escape into the room.

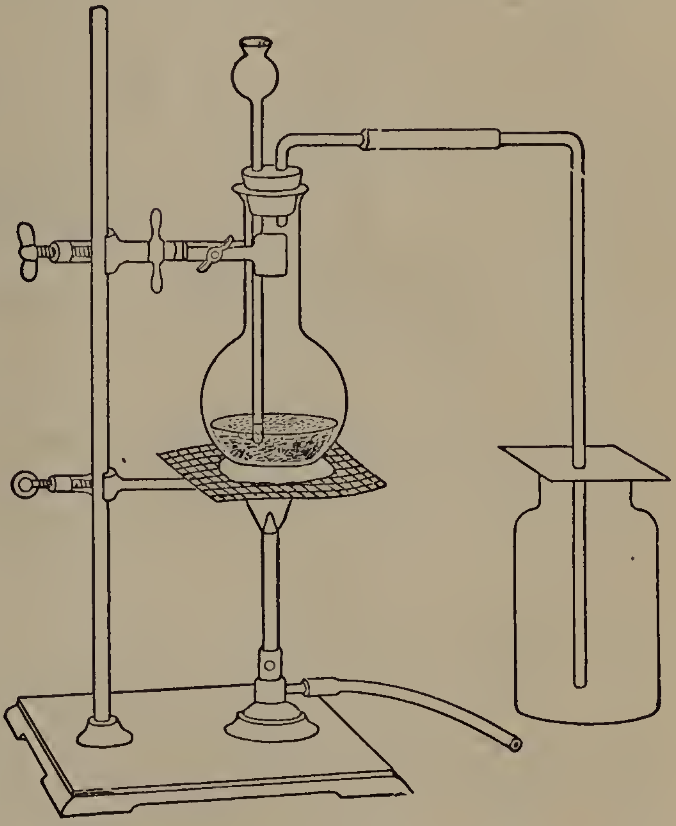

Fig. 41. Preparing hydrogen chloride.

b. Properties. Invert a bottle of the gas in a dish of water, remove the glass plate, and *note what happens.*

Test the gas in another bottle with a lighted splint. *Is it combustible? Is it a supporter of combustion?*

Pour a little ammonium hydroxide on a wad of filter paper and throw it into a bottle of hydrogen chloride. *What is the result?*

c. **Hydrochloric acid** is the water solution of hydrogen chloride. Dip a stirring rod into the solution and apply a drop to red and blue litmus papers.

Pour some of the hydrochloric acid just made into a test tube and drop into it a strip of magnesium. Test the gas set free with a flame. *Result? Write the equation for this reaction.*

Fig. 42. Testing for hydrogen chloride with a drop of ammonium hydroxide.

Optional work. Place in separate tubes small quantities of the following chlorides: **potassium chloride** (KCl), **ammonium chloride** (NH_4Cl), **calcium chloride** ($CaCl_2$), and **ferric chloride** ($FeCl_3$). Add a few drops of concentrated sulfuric acid (H_2SO_4) and, if necessary, warm gently to start the reaction. *Is the material boiling?* Waft a little of the gas toward the nose. *What is the odor?* Blow across the mouth of each tube. *How does this gas behave with water vapor?* Place moistened blue litmus paper across the mouth of each test tube. *What does this show about the aqueous solution of the gas?* Dip a glass rod in ammonium hydroxide solution (NH_4OH); now insert the glass rod into the mouth of the test tube (Fig. 42). The product is ammonium chloride (NH_4Cl).

EXPERIMENT 18

TEST FOR A CHLORIDE

Apparatus. — Test tubes and rack.

Materials. — Dilute hydrochloric acid. Dilute nitric acid. Solutions of silver nitrate (5%), ammonium hydroxide, sodium phosphate, and potassium oxalate. " Unknown " solution.

a. Fill a test tube half full of dilute hydrochloric acid and add a few drops of silver nitrate solution ($AgNO_3$). The solid which separates out, *i.e.* the **precipitate**, is silver chloride (AgCl). a white powder

Record the color and appearance of this precipitate. Label and set the tube aside for further experimenting.

b. In the same way add a few drops of silver nitrate solution to a solution of sodium phosphate. The precipitate in this case is silver phosphate. [redacted] yellow

Record its color and appearance. Label the test tube and set it aside.

ammonium

c. To a solution of potassium oxalate add a few drops of silver nitrate solution. The precipitate is silver oxalate. white

Record its color and appearance. Label this test tube and set it aside. [redacted] curd.

d. Try the effect of dilute nitric acid on each of the three precipitates just prepared. *How can you distinguish between silver chloride, silver phosphate, and silver oxalate?* a — no, c, yes
b — yes.

e. Prepare another portion of silver chloride by adding silver nitrate solution to a solution of sodium chloride (common salt). *Find out whether this precipitate is soluble in ammonium hydroxide.* Yeeeeees

f. Prepare still another portion of silver chloride and stand the tube in the sunlight. *What is the result?*

Summarize the three characteristic properties of silver chloride. How would you test for a soluble chloride?

g. Apply these tests to an " unknown solution " which you may obtain from the instructor. In making your report on this unknown solution, record the result of each step in the process of testing as well as your final conclusion.

EXPERIMENT 19

Acids, Bases, and Salts

Apparatus. — Test tubes and rack. Stirring rod. Evaporating dish. Tripod and asbestos gauze. Glass plate. Bunsen burner.

Materials. — Dilute hydrochloric, sulfuric, nitric, and acetic acids. Litmus papers (red and blue). Solutions of sodium hydroxide, potassium hydroxide, ammonium hydroxide, and calcium hydroxide. Sodium hydroxide (sticks).

a. Acids. Prepare dilute solutions of the following acids by adding a few drops of the acid to 10 cc. of water: hydrochloric, sulfuric, nitric, and acetic. Lay a piece of blue litmus paper and a piece of red litmus paper on a glass plate. Touch both kinds of paper with the end of a clean glass rod wet with each acid in turn. *What changes do you observe in each case?* red

Taste *one* drop of each of the dilute solutions and immediately after tasting rinse the mouth with water. *What is the distinctive taste of acid?* very sour.

Write the formulas of these acids (see textbook) *and find out what element is present in all of them.*

b. Bases. In a similar way test with litmus papers dilute solutions of the following bases: sodium hydroxide, potassium hydroxide, ammonium hydroxide, and calcium hydroxide. *What is the characteristic change in litmus which you observe with bases?*

Taste a drop of calcium hydroxide solution. *How would you characterize the taste of bases?* biting

Write out the formulas of these bases and find out in what respect they are similar in composition.

c. Salts. Dissolve a small piece of sodium hydroxide (a stick about 1 cm. long) in about 20 cc. of water in a test tube. Stir it until all is dissolved and then pour out about 15 cc. of the solution into an evaporating dish (Fig. 43). Add dilute hydrochloric acid a little at a time and keep stirring the liquid until a drop on a glass rod gives no basic reaction with litmus.

Then dilute the 5 cc. of sodium hydroxide solution which was left in the test tube until it is five times as dilute, and also dilute the acid solution the same amount. Now use these much diluted acid and base solutions to bring the solution in the evaporating dish to the neutral point, or **end point,** *i.e.* the point where it affects neither red nor blue litmus paper. This process is called **neutralization.**


Fig. 43. **Neutralizing a base with an acid.**

Evaporate the neutral solution to dryness slowly so that the liquid does not spatter. Taste the residue. *What is it?* Salt.

The scientific name for the residue is sodium chloride, which indicates that it contains only sodium and chlorine. *What has probably become of the other elements in the acid and base?* gone into the atmosphere.

Optional work. The process in part (c) is a general method of preparing neutral salts. Prepare other salts, such as potassium nitrate (KNO_3), sodium sulfate (Na_2SO_4), or ammonium chloride (NH_4Cl).

EXPERIMENT 20

DETERMINATION OF THE CONCENTRATION OF A SOLUTION BY TITRATION

Given a fifth-normal solution of sodium hydroxide, to find the concentration of a hydrochloric acid solution by neutralization, using burettes.

Apparatus. — Two burettes. Small Erlenmeyer flask or beaker. Stirring rod. Ring stand with 2 clamps.

Materials. — Fifth-normal solution of sodium hydroxide and a solution of hydrochloric acid (nearly the same concentration). Phenolphthalein solution (1 g. dissolved in 200 cc. of alcohol).

a. A **normal solution of a base** contains 17 g. of replaceable hydroxyl (OH) per liter. *How many grams of sodium hydroxide ($NaOH$) must be dissolved in water and made up to a liter to form a fifth-normal* (0.2 *N*) *solution?*

A **normal solution of an acid** contains 1 g. of replaceable hydrogen per liter. *How many grams of hydrogen chloride (HCl) does one liter of a normal hydrochloric acid solution contain?*

b. Fill one burette nearly full of a sodium hydroxide solution of known concentration (0.2 N). Label the burette and clamp it in a vertical position. Then fill another burette with the hydrochloric acid whose concentration is to be determined (Fig. 44). Draw off from each burette enough liquid to remove the air bubbles from the tip and to bring the **meniscus** (the curved surface of the liquid) down to the graduated portion of the burette.

Examine carefully the markings on the burettes so as to understand the graduations. In reading a burette, make sure that the eye is on a level with the lowest part of the meniscus, as shown in figure 12. *Record the readings of both burettes in tabular form as given below.*

NOTE. Observe that the graduation is downwards. If then the reading is four-tenths of a cc. above the 1 cc. mark, the reading is 0.6 cc. (not 1.4 cc.).

Let about 10 cc. of the basic solution flow out into a little flask and then add a drop or two of some indicator, such as **phenolphthalein solution.** Remember that this indicator turns a pink color when the solution is basic ; an acid solution remains colorless.

Now set the flask on a sheet of white paper under the other burette and allow the acid to run in a few drops at a time. Shake the solution in the flask vigorously after each addition of a few drops. When the acid begins to decolorize the indicator near the point where it enters, work more cautiously. Add one drop at a time and shake up the solution until the pink color just disappears. Now add a drop or two of the base until the reddish tinge just appears ; see if one drop of acid will make the solution colorless. Continue in this way until a reversal of color is got by *one drop* of base or acid.

Read the level of the liquid in each burette to one-tenth of a cubic centimeter (0.1 cc.) and record in the table. To find the volume of base used, subtract the first burette reading (before neutralization) from the burette reading after neutralization and record that result in the column marked Difference in the table. In a similar way find the volume of acid used to neutralize the base. Since the volumes of base and acid used are *inversely* proportional to the concentrations of the base and acid, we can easily compute the concentration of the acid (assuming the base to be 0.2 N).

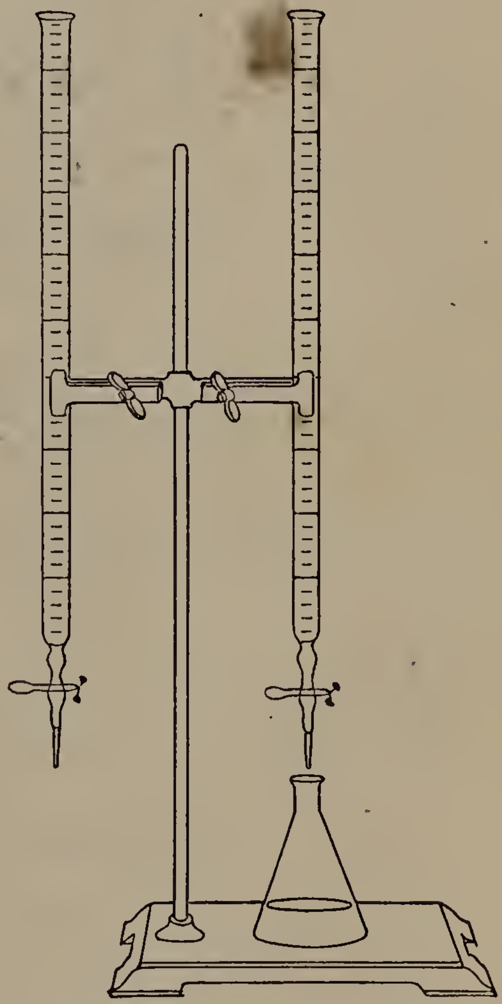
Fig. 44. Titration of a base with an acid.

Wash out the flask and make another determination; repeat the process until you get three consistent results. Take the average, or mean, of these three results as the concentration of the acid.

TABLE

Titration	[redacted] ACID		[redacted] BASE	
	Readings	Difference	Readings	Difference
No. 1	0 - 16.4	16.4	12.8	12.8
2	16.4 - 33.2	16.8	12.8 - 25.6	12.8
3	33.2 - 40.5	7.3	25.6 - 30.8	5.2

Optional work. We may determine in this way the concentration of any base, such as **household ammonia**, if we have a standard or known acid solution. But if we wish to determine the amount of acid, in such substances as **vinegar, cider, or lime juice**, we must have a standard base solution.

EXPERIMENT 21

PREPARATION OF SOLUBLE SALTS

Apparatus. — Test tubes and rack. Tripod and asbestos gauze. Evaporating dish. Bunsen burner. Funnel. Stirring rod.

Materials. — Dilute hydrochloric, nitric and sulfuric acids. Zinc. Magnesium oxide. Sodium carbonate. Dilute solutions of sodium hydroxide and potassium hydroxide. Litmus papers. Filter paper.

a. Metal and acid. Many of the soluble salts (see Table of Solubilities in Appendix) of the common metals, such as zinc (Zn), iron (Fe), magnesium (Mg), and copper (Cu), can be prepared by replacing the hydrogen in the common acids. Such salts as zinc chloride ($ZnCl_2$), magnesium sulfate

($MgSO_4$), copper nitrate ($Cu(NO_3)_2$), and iron sulfate ($FeSO_4$) can be thus prepared.

To 5 cc. of dilute hydrochloric acid add zinc until there is no further action. Filter, evaporate the filtrate almost to dryness, and cool. *What is the residue? Write the equation.*

b. Metallic oxide and acid. Dissolve about 5 g. of magnesium oxide (MgO) in dilute sulfuric acid and filter if necessary; then evaporate the clear solution to a small volume and cool. *What is the residue? Write the equation.*

c. Carbonate and acid. Heat 10 cc. of a solution of sodium carbonate (Na_2CO_3) to the boiling point and add dilute hydrochloric acid as long as it causes effervescence. Evaporate the resulting solution to dryness. *What is the residue? Write the equation.*

Try the action of hydrochloric acid on solid sodium carbonate. *Compare the action of hydrochloric acid on a carbonate with the action of sulfuric acid on a chloride.*

d. Neutralization of a base and an acid. Sodium chloride ($NaCl$) has already (Exp. 19) been prepared by neutralizing sodium hydroxide ($NaOH$) with hydrochloric acid. In a similar way potassium chloride (KCl), sodium sulfate (Na_2SO_4), potassium sulfate (K_2SO_4), sodium nitrate ($NaNO_3$), and potassium nitrate (KNO_3) can be prepared.

Make one of these salts by neutralizing a suitable base and acid. Evaporate the salt solution nearly to dryness and examine the residue. *Write the equation.*

$ZnCl_2 + H_2SO_4 \rightarrow$ [illegible]

EXPERIMENT 22

Preparation of Insoluble Salts

Apparatus. — Test tubes and rack. Small beakers. Asbestos gauze. Bunsen burner. Tripod.

Materials. — Solutions of lead nitrate and lead acetate. Lead oxide (litharge) and lead carbonate. Sodium chloride solution. Dilute hydrochloric acid.

We may illustrate the various ways of preparing an insoluble salt by preparing lead chloride ($PbCl_2$) as follows:

a. Salt and acid. Heat in a test tube 20 cc. of lead nitrate ($Pb(NO_3)_2$) solution to boiling and add 5 cc. of dilute hydrochloric acid. Set aside to cool. *What is the precipitate? Write the equation.* lead chloride. white.

b. Salt and salt. Heat 20 cc. of lead acetate ($Pb(C_2H_3O_2)_2$) solution and add 5 cc. of sodium chloride solution. Set aside to cool. *What is the precipitate? Write the equation.*

c. Oxide and acid. Dissolve in a beaker 1 g. of lead oxide (PbO) in boiling dilute hydrochloric acid and add about 50 cc. of boiling water. Decant the clear solution and set it aside. *What is the precipitate? Write the equation.*

d. Salt and acid. Dissolve 1 g. of lead carbonate ($PbCO_3$) in boiling hydrochloric acid and proceed as in part (**c**).

Optional work. Devise methods of preparing barium sulfate ($BaSO_4$), lead sulfate ($PbSO_4$), silver chloride ($AgCl$), and lead iodide (PbI_2). Prepare one of these salts in as many ways as your time permits and submit samples. Find out how this salt is prepared commercially and why.

60 cents

EXPERIMENT 23

GRAM-MOLECULAR WEIGHT OF CARBON DIOXIDE

To find weight in grams of 22.4 liters of carbon dioxide

Apparatus. — A 500-cc. flask with 1-hole stopper. Pinchcock. Rubber tube. Two wide-mouth bottles with 2-hole stoppers. Thistle tube. Horn pan balance and weights. Ring stand. Glass tubing. Thermometer. Barometer.

Materials. — Marble chips. Dilute hydrochloric acid. Concentrated sulfuric acid.

a. When the chemist uses as many grams of a substance as there are units in its molecular weight, he calls this weight the **gram-molecular weight.** Experiments show [§ **137**] that *the gram-molecular weight of any gas occupies under standard conditions 22.4 liters.* In the laboratory we weigh a given volume of gas (reduced to standard conditions) and then compute the weight in grams of 22.4 liters.

b. Arrange a large flask with a 1-hole rubber stopper which has a glass tube extending nearly to the bottom of the flask. Attach to the outer end of the glass tube a short rubber tube provided with a pinchcock. Weigh the flask fitted with the stopper, tube, and pinchcock with great care to a centigram (0.01 g.). Then open the pinchcock and connect the rubber tube to a supply of dry carbon dioxide. This can easily be obtained by putting a few pieces of marble in a generating bottle and connecting it with a gas-washing bottle which contains concentrated sulfuric acid, as shown in figure 45.

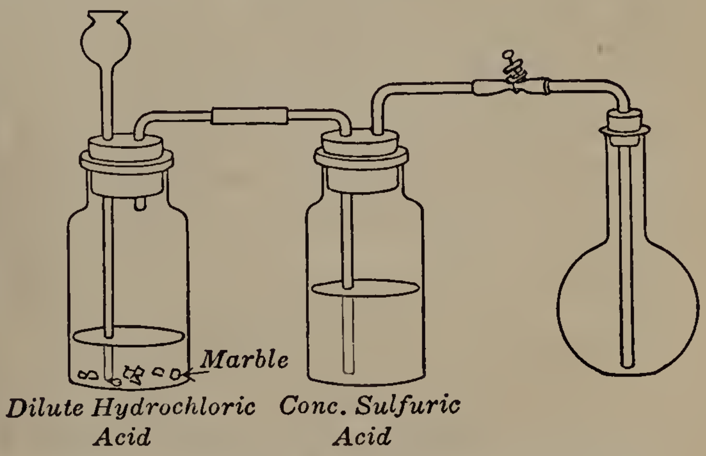

Fig. 45. **Filling a flask with dry carbon dioxide.**

c. Add dilute hydrochloric acid to the marble, loosen the rubber stopper in the flask so that a free current of gas may pass through, and pass a steady stream of carbon dioxide through the apparatus for 10 minutes. Then insert the stopper as before and disconnect from the generator. Open the pinchcock for an instant so that the pressure inside the flask may be the same as outside, and weigh the flask to centigrams.

To make sure that the flask is entirely filled with carbon dioxide, it is well to connect it again with the generator and let the gas pass through for 5 minutes longer. As soon as constant weight has been attained (which indicates that the flask is entirely filled), read and record the temperature of the room and the barometric pressure.

d. The gain in weight of the flask when filled with carbon dioxide is, of course, only a small quantity because it is the difference between the weight of the carbon dioxide and the weight of the air in the flask. To find the weight of the air, we determine its volume by filling it with water from a graduated cylinder, reducing this volume to standard conditions, and then computing the weight of that volume of air. (Assume that 1 liter of air at 0° C. and 760 mm. weighs 1.29 g.) To get the weight of the carbon dioxide in the flask, add the weight of the air in the flask to the gain in weight when filled with carbon dioxide. We now have determined the weight of a certain volume (reduced to standard conditions) of carbon dioxide and can readily compute the weight of 22.4 liters of the gas.

Arrange your calculations in an orderly fashion and record your data and results in tabular form:

Weight of flask with air . . . . g.
Weight of flask with carbon dioxide, 1st trial . . . . g.
Weight of flask with carbon dioxide, 2d trial . . . . g.
Gain in weight of flask when filled with carbon dioxide g.
Temperature of room . . . . ° C.
Barometric pressure . . . . mm.

Volume of the flask . . . . cc.
Volume of flask at 0° C. and 760 mm. . . . . cc.
Weight of air in flask (1 liter = 1.29 g.) . . . . . . g.
Weight of carbon dioxide in flask . . . . g.
Weight of 22.4 liters of carbon dioxide . . . . g.

Problems. **1.** Assuming one liter of air weighs 1.29 g., compute the weight of 22.4 liters.

2. From the answer to problem 1 and the result of this experiment, compute the relative heaviness of carbon dioxide referred to air; also compute how many times heavier carbon dioxide is than hydrogen.

3. Assuming the formula of carbon monoxide to be CO and its molecular weight to be $12+16$ or 28, compute its gram-molecular weight and its relative heaviness referred to air.

4. The density of hydrogen chloride gas is 1.64 grams per liter, and the percentage composition is 2.8% hydrogen and 97.2% chlorine. Compute its gram-molecular weight and its formula.

EXPERIMENT 24

ATOMIC WEIGHT OF COPPER

To find the combining weight of copper and the factor by which this weight must be multiplied in order to get the atomic weight.

Apparatus. — Ring stand and clamp. Ignition tube (hard-glass test tube). Horn pan balance and weights. Bunsen burner. Glass tubing. Rubber tubing.

Materials. — Copper oxide (powder).

a. Preliminary statement. First we may determine the combining weight of copper by reducing a known weight of copper oxide to metallic copper. Assuming the combining weight of oxygen to be 8, we may compute from the actual weights of copper and oxygen, the **combining weight of copper.** The atomic weight of copper is either equal to or some multiple of its combining weight. To determine this multiple we may make use of a fact discovered by Dulong and Petit: *the products* *of the atomic weights of the solid elements by their specific heats* are in almost all cases numbers lying between* 6 *and* 7, *the average being* 6.4. This is *not* a method of *determining* atomic weights but is an easy way to find the factor by which the combining weight must be multiplied in order to get the atomic weight.

b. Procedure. Clean and dry a hard-glass test tube (ignition tube) and weigh it to a centigram (0.01 g.). Slide into the tube by means of a folded strip of paper 2 or 3 g. of black copper oxide (powder). Get all the oxide at the bottom of the tube and carefully weigh the tube with the copper oxide. Clamp a piece of glass tubing in a horizontal position as shown in figure 46. Since illuminating gas contains a large percentage of hydrogen, it is convenient to use it as the reducing agent. We may burn the excess of gas as it issues from the mouth of the test tube and then turn the gas down so that the flame is only about 1 cm. high. Heat the test tube slowly until the oxide is red-hot. After 5 minutes' heating, turn off the gas and rotate the test tube, tapping the test tube to make the oxide fall to the other side. Again insert the gas tube and continue heating until the black copper oxide is entirely reduced to red copper. Cool the ignition tube and weigh again.

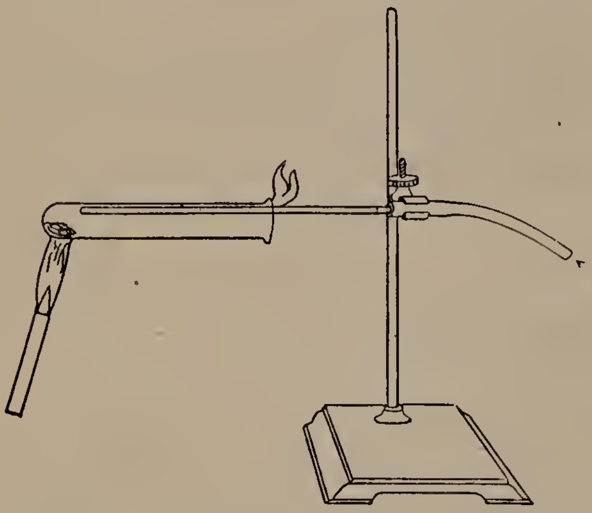
Fig. 46. Reducing copper oxide to copper with illuminating gas.

c. Computation. The loss of weight is the weight of the oxygen removed from the oxide. Assuming the combining

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*Heat is measured in calories. A **calorie** is the heat required to raise the temperature of a gram of water 1° C. The number of calories required to raise the temperature of a gram of a substance 1° C. is called its **specific heat**.

weight of oxygen to be 8, *calculate the combining weight of copper;* thus:

$$\text{Wt. of oxygen : wt. of copper : : } 8 : x.$$

Assume the specific heat of copper to be 0.0936 and the constant product (at. wt. × sp. ht.) to be 6.4; *compute the approximate atomic weight of copper. What is the factor* (whole number) *by which the combining weight must be multiplied in order to get the atomic weight?* Multiply the combining weight of copper by this factor, and *the product is the atomic weight.* Compare this result with the value given in the Table of Atomic Weights and *compute the percentage error.*

Show clearly all your computations and record your data and results in tabular form.

Wt. of test tube . . . . g.
Wt. of test tube + copper oxide . . . . g.
Wt. of test tube + copper . . . . g.
Wt. of oxygen . . . . g.
Wt. of copper . . . . g.
Combining wt. of copper (Oxygen = 8) . . . .
Factor by which the combining wt. must be multiplied in order to get the atomic wt. . . . .
Atomic wt. of copper . . . .
Atomic wt. of copper according to Table of Atomic Weights
Percentage error . . . . %

Optional work. To determine the atomic weight of iron, change about 2 g. of iron wire (piano wire) into the oxide by dissolving it in nitric acid and then evaporating the solution to dryness over a water bath and heating directly but carefully as long as any red fumes are given off. Finally heat to constant weight. The gain in weight is the weight of the oxygen combined with the known weight of iron. Assuming the combining weight of oxygen to be 8, *compute the combining weight of iron.* Taking the specific heat of iron as 0.119, *calculate the atomic weight of iron. Calculate the formula of this oxide of iron.*

EXPERIMENT 25

Solutions of Gases, Liquids, and Solids

Apparatus. — Test tubes and rack. Bunsen burner. Funnel. Watch glasses.

Materials. — Concentrated ammonium hydroxide. Carbon tetrachloride. Glycerin. Copper sulfate. Potassium chlorate. Calcium carbonate (powdered). Filter papers.

a. Gases in water. Warm gently in a test tube a little water drawn from the faucet. *Is there any evidence of a gas having been dissolved in the water?*

In the same way heat gently a little ammonia water (ammonium hydroxide) and note any evidence of a gas having been dissolved in the water.

Are gases more soluble in hot or in cold water?

b. Liquids in liquids. Add to 5 cc. of water in a test tube 5 cc. of carbon tetrachloride, a few drops at a time, and shake the mixture vigorously after each addition. *Are water and carbon tetrachloride miscible at each stage?*

Repeat, using glycerin and water.

c. Solids in liquids. Place in separate test tubes about 1 g. of each of the following powdered substances: copper sulfate ($CuSO_4$), potassium chlorate ($KClO_3$), and calcium carbonate ($CaCO_3$). Add to each substance 10 cc. of water and shake each test tube.

What evidence is there that these substances have dissolved?

If there is any doubt as to whether any of a solid has dissolved, filter off 5 cc. of the liquid and evaporate on a watch glass to dryness.

If in any case all the solid has dissolved, add another gram of the powdered solid and continue this process as long as it is completely dissolved.

What are the relative solubilities of these three substances in water?

Optional work. What are the best conditions for the solution of a solid? Study the **effect of surface.** Take two crystals of copper sulfate of about the same size, each of which can be slipped into a test tube. Grind one of the crystals to a fine powder in a mortar and place the powder in a test tube. In another test tube place the other crystal. Add 20 cc. of water to each test tube; cork and shake the tubes gently. Determine how long it takes the powder to dissolve and what fraction of the original crystal still remains in the tube. *Why does pulverizing hasten the process of solution?*

EXPERIMENT 26

Effect of Temperature on Solubility

Apparatus. — Test tubes and rack. Ring stand. Bunsen burner. Asbestos gauze. Large beaker.

Materials. — Copper sulfate. Sodium chloride.

a. Solubility in cold water. Add to 10 cc. of water in a test tube 2 g. of well-pulverized copper sulfate and shake the tube until the solid dissolves. Continue adding 2-gram portions until the solution is saturated, that is, until some of the solid remains undissolved after shaking. *Calculate the amount* (g.) *which would dissolve in* 100 *g. of cold water.*

Repeat the experiment, using sodium chloride (common salt) instead of copper sulfate.

b. Solubility in hot water. Heat the solutions just prepared in a beaker of boiling water (Fig. 47) and continue to add 2-gram portions of the solid until each solution is saturated at 100° C. *Compute for each solid the amount which dissolves in* 100 *g. of hot water.*

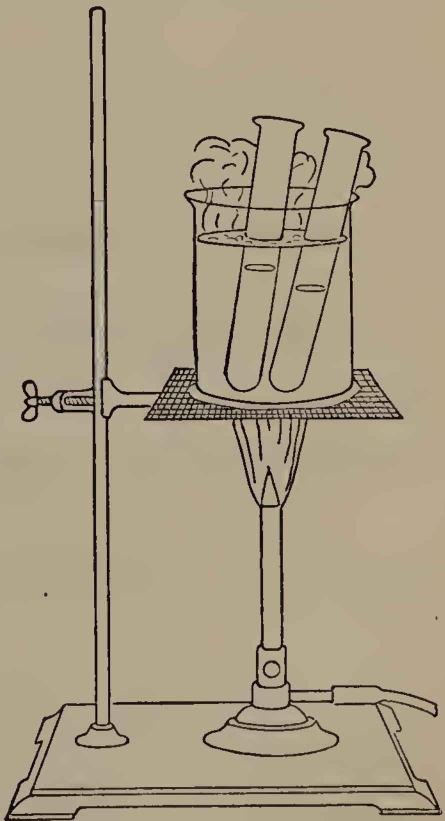

Fig. 47. Heating solutions in hot water.

c. Cooling a saturated solution. Cool the solutions just prepared (**b**) by holding the test tubes in running water from the faucet. *Observe the amount and size of the crystals formed in each tube.*

Warm the contents of the tubes carefully by putting them once more into the boiling water until the crystals are again dissolved. Set the tubes away in the test-tube rack and let them cool slowly until the next laboratory period. *Compare the crystals formed quietly and slowly with those formed quickly.*

Optional work. Supersaturation. Make a saturated solution of sodium sulfate crystals by shaking the salt with water at 33° C. Reheat the solution if it falls below that point (**transition temperature**). Pour into three test tubes clear portions of the solution. Cool the tubes in cold water without shaking. The solutions should now contain no crystals.

Put into one portion a very small crystal of the salt; into another portion a bit of sodium sulfate which has been dehydrated by heating on a glass rod; and shake the third portion violently. *Compare the results of these experiments.*

EXPERIMENT 27

SOLUBILITY OF COMMON SALT

To find how many grams of common salt will dissolve in 100 *grams of water.*

Apparatus. — Small flask with stopper. Evaporating dish. Horn pan balance and weights. Ring stand. Copper water bath or large beaker. Tripod. Bunsen burner. Thermometer. Funnel.

Materials. — Sodium chloride. Filter paper.

a. First make a saturated solution of sodium chloride. This can be done by placing 10 g. of common salt with 20 cc. of water in a stoppered flask or bottle and shaking the contents at intervals for 15 minutes; *or* by dissolving 10 g. of salt in 20 cc. of boiling water and allowing the solution to cool to the temperature of the room (shaking the solution occasionally). In either case some of the salt should remain undissolved.

b. Take the temperature of the saturated salt solution just prepared. Carefully weigh a clean, dry evaporating dish and then pour into it through a filter a portion of the solution. Reweigh as rapidly as possible, out carefully.

c. Evaporate the solution completely to dryness upon a water bath (Fig. 48) or on a beaker of boiling water (Fig. 49). Be careful not to lose any of the salt by spurting. Let the dish and contents cool and again weigh. Reheat the dish and contents until the weight is constant.

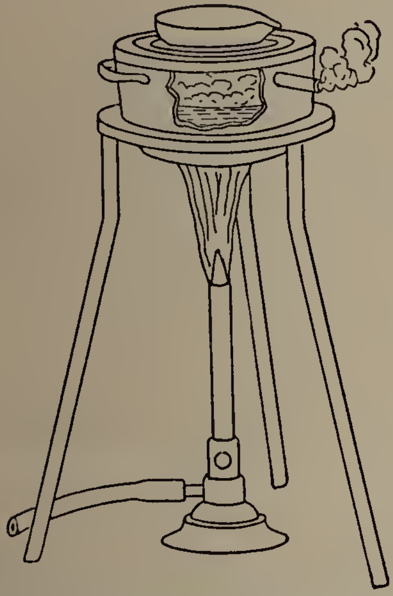

Fig. 48. **Evaporating over a water bath (copper).**

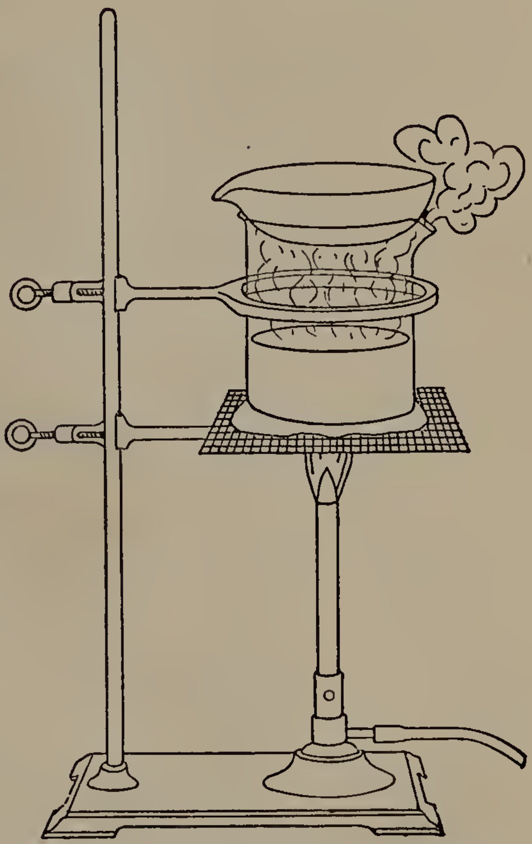

Fig. 49. **Evaporating on a beaker of boiling water.**

Note. Care must be taken to replace from time to time the water in the copper bath or beaker.

d. *Compute the number of grams of salt soluble in 100 g. of water at the observed temperature.* Compare your result with the results plotted in the curves shown in figure 86 on page 152 of the textbook. Record your data and results in tabular form somewhat as follows:

Temperature of solution . . . . °C.
Wt. of dish + solution . . . . g.
Wt. of dish . . . . g.
Wt. of solution . . . . g.
Wt. of dish + dry solid . . . . g.
Wt. of dish . . . . g.
Wt. of salt . . . . g.
Wt. of water . . . . g.
Wt. of salt dissolved in 100 g. of water at °C. . . . . . g.

Optional work. The same method can be applied to determine the solubility of copper sulfate ($CuSO_4 \cdot 5\ H_2O$) or potassium dichromate ($K_2Cr_2O_7$) at various temperatures.

EXPERIMENT 28

WATER OF CRYSTALLIZATION — EFFLORESCENCE — DELIQUESCENCE

Apparatus. — Three watch glasses. Ring stand with clamp. Bunsen burner. Test tubes.

Materials. — Crystals of sodium carbonate and gypsum. Fused calcium chloride. Crystals of potassium chlorate, potassium nitrate, potassium alum, zinc sulfate, sodium sulfate, and copper sulfate.

a. Place a fresh glassy crystal of washing soda (sodium carbonate) on a watch glass and set it aside until the next laboratory period. Treat in the same way a small crystal of gypsum and a piece of fused calcium chloride. *After a day or two describe any changes which you observe in the appearance of each substance.*

NOTE. Sodium carbonate crystals are **efflorescent** in air which contains the average amount of moisture.

Calcium chloride is **deliquescent** under the same conditions.

b. Place a clear crystal of washing soda in a dry test tube and clamp the tube in a horizontal position. Heat the crystal gently and observe any changes in its appearance. *What condenses on the walls of the tube?*

c. In separate dry test tubes warm gently (Fig. 50) a few crystals of each of the following substances: potassium chlorate, potassium nitrate, potassium alum, zinc sulfate, and sodium sulfate.

Record the results in tabular form as follows:

SUBSTANCE HEATED	AMOUNT OF CONDENSED WATER	APPEARANCE OF RESIDUE

NOTE. The amount of condensed water may be indicated by the words, "a trace," "little," or "much." Even anhydrous substances often contain a little moisture, which is said to be "water mechanically held."

Are all crystalline substances hydrates?

d. In another test tube heat gently a small crystal of copper sulfate (blue vitriol). *Observe the change in color and form.*

When the tube is cold add a few drops of hot water. *Note the change in color.* Pour the solution on a watch glass and set it aside to recrystallize. *Compare the product with the original substance.*

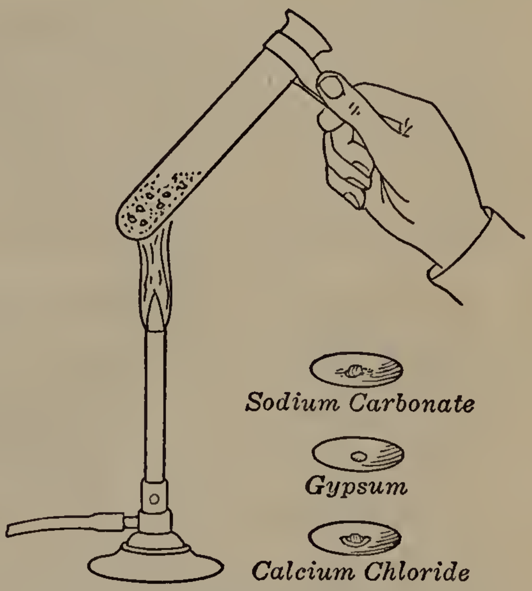

Fig. 50. Testing substances for water of crystallization.

Optional work. Test a small crystal of cobalt chloride (about 5 mm. across) in the same way that you did copper sulfate in (**d**). The solution is called "**sympathetic ink.**" Use a clean pen to write with cobalt chloride solution on paper. The writing while slightly moist is almost invisible, but it comes out when dried over a low flame.

EXPERIMENT 29

MEASUREMENT OF WATER OF CRYSTALLIZATION

To find the per cent of water of hydration in gypsum.

Apparatus. — Tripod. Pipe-stem triangle. Porcelain crucible. Horn pan balance and weights. Bunsen burner. Ring stand.
Materials. — Gypsum crystals.

a. The amount of crystal water contained in a hydrate, such as gypsum, is found by heating a known weight of the crystallized substance until all the water is driven off. The process is completed when the substance ceases to lose weight on heating.

b. Weigh a clean, dry crucible with cover to the nearest centigram (0.01 g.). Place about 5 g. of pulverized gypsum (do not try to take this exact amount) in the crucible and weigh again with cover carefully. Record these weights in tabular form as shown below.

c. Place the covered crucible on the pipe-stem triangle and place this on the tripod. Heat the crucible at first very gently, holding the burner in the hand; then place the burner under the crucible and adjust the flame so that the top of the inner blue cone is a short distance below the crucible. After heating for 10 minutes, remove the cover and continue heating strongly for 5 minutes longer. Then *gradually* cool the crucible and reweigh to the nearest centigram.

d. Reheat the crucible (without cover) for 5 minutes longer, then allow to cool, and reweigh. Repeat this operation until the weight is constant. From your results calculate the percentage of water of hydration present in the crystals.

Wt. of crucible + gypsum . . . . g.
Wt. of crucible empty . . . . g.
Wt. of gypsum taken . . . . g.
Wt. after first heating . . . . g.

Wt. after last heating . . . . ____ g.

Wt. of water of hydration . . . . g.

Percentage of water $= \dfrac{\text{wt. of water}}{\text{wt. of gypsum}} =$ $=$ %

Optional work. Calculate from the results of this experiment the number of molecules of water in a molecule of gypsum. Assume the anhydrous substance is $CaSO_4$ and use the approximate values for the atomic weights (Table facing front cover) to compute the molecular weights. Write the formula for gypsum.

EXPERIMENT 30

ELECTRICAL CONDUCTIVITY OF SUBSTANCES IN SOLUTION

Apparatus. — Conductivity apparatus as described below (Fig. 51). Electric current from lighting system or battery.

Materials. — Solutions of hydrochloric acid, sodium chloride, sodium hydroxide, alcohol, sugar, acetic acid, and ammonium hydroxide.

a. Apparatus. If the laboratory is provided with electric lights (110 volts) suspended over the tables, it is easy to remove a lamp bulb and insert in its place a plug with extension cord. Connect the electrodes of your conductivity apparatus (Fig. 51) in series with an electric light socket. Screw a lamp (20–40 watt) into the socket of your apparatus and test your electric circuit by short-circuiting the two electrodes with a knife-blade. The lamp should glow brightly; in fact the brilliancy of its light will measure roughly the electrical conductivity of the short-circuiting substance. Before testing the conductivity of any given solution rinse the electrodes with distilled water until, when they are held for

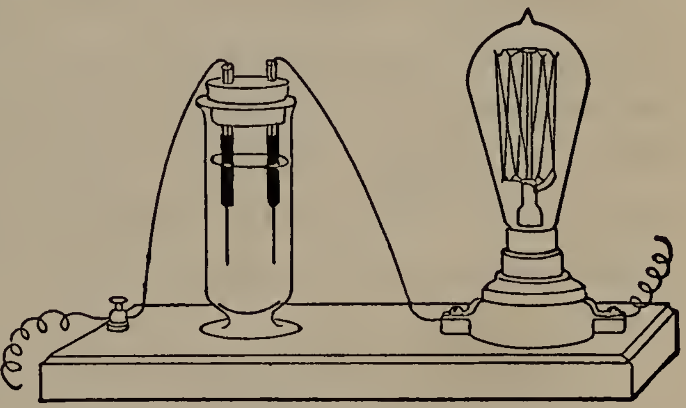

Fig. 51. Testing the conductivity of solutions.

10 seconds or more immersed in distilled water, no sign of a glow is seen in the lamp filament.

NOTE. In case the platinum electrodes are not available, it is possible to use stout copper-wire electrodes, provided they are polished bright with emery paper and kept clean and bright. A glass tumbler or wide-mouth bottle will serve to hold the electrolyte.

In cases where the electric lighting circuit can not be used, 4 dry cells or a 6-volt storage battery with a 6-volt miniature lamp as an indicator can be employed.

b. Electrolytes and nonelectrolytes. In setting up your conductivity apparatus and every time you change the solution, *loosen the lamp in the socket of your apparatus and do not screw it down to make contact until all the connections have been made.* Test the conductivity of the following solutions: (1) dilute hydrochloric acid (1 : 4); (2) sodium chloride (10%); (3) sodium hydroxide (10%); (4) alcohol (10%); (5) sugar (10%); (6) distilled water.

Classify the substances used above according to their electrical conductivities.

c. Relative conductivity of acids. Prepare solutions of hydrochloric acid and acetic acid which have the same molecular concentration, for example one-fifth normal (0.2 N). *Compare the conductivity of these acid solutions as indicated by the brightness of the lamp. Why is it necessary to have the electrodes immersed to the same depth in both cases? Which acid is more completely ionized?*

d. Relative conductivity of bases. Prepare solutions of sodium hydroxide and ammonium hydroxide which have the same molecular concentration, for example one-fifth normal (0.2 N). *Compare the conductivity of these basic solutions as indicated by the brightness of the lamp.* Have the electrodes immersed to the same depth in each case. *Which base is more completely ionized?*

e. Ions of acids and bases. *What ion is common to all acids? What is the other ion of hydrochloric acid? of acetic acid?*

What ion is common to all bases? What is the other ion of sodium hydroxide? of ammonium hydroxide?

What is the significance of the terms "strong" and "weak" as applied to acids and bases?

Optional work. To illustrate the electrolysis of a salt solution, first plug the bottom of a U-tube with absorbent cotton to hinder the rapid diffusion of the liquid. Then nearly fill the U-tube with sodium sulfate (Na_2SO_4) solution (about 15%) which is colored with a neutral litmus solution. Clamp the tube in an upright position and insert a platinum electrode in each arm so that it is just immersed in the solution (Fig. 52). The electrodes may be of platinum foil or a spiral of platinum wire. Let the current from several dry cells or a storage battery flow through the solution until there is a decided change in color of the litmus solution at the two electrodes. (See B. & C. p. 10.)

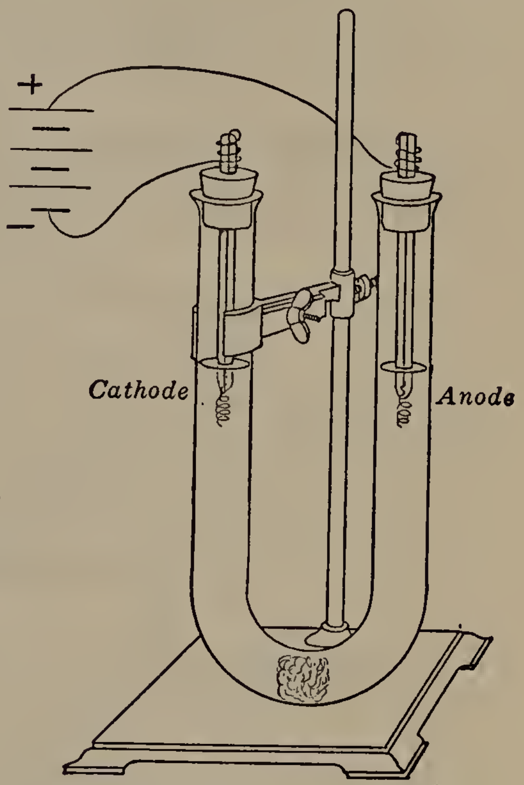

Fig. 52. Electrolysis of a salt solution.

What gas is evolved at the negative electrode (cathode)? What gas is evolved at the positive electrode (anode)? How do you account for the change in color at the electrodes? Write the equations for the chemical changes involved.

EXPERIMENT 31

Boiling Points of Solutions

Apparatus. — Ring stand with one ring and clamp. Asbestos gauze. Bunsen burner. Boiling flask (250 cc.) with side tube. Wide-mouth bottle. Sheet of cardboard. Thermometer.

Materials. — Alcohol. Salt solutions (10 g. in 100 cc. water and 20 g. in 100 cc. water). Sugar solution (20 g. in 100 cc. water).

a. Water. In order to show the effect of various substances on the boiling point of water, it is necessary first to determine the boiling point of pure water. If the atmospheric pressure happened to be 760 mm., and if there were no error in the thermometer used, we should expect a thermometer placed in boiling water to indicate 100° on the Centigrade scale. But as it is hardly likely that both of these conditions obtain, we shall first arrange the apparatus as shown in figure 53 and then place a small quantity (4 or 5 cm. deep) of distilled water in the boiling flask. It is well to have the thermometer bulb just immersed in the water. When the Bunsen burner is placed underneath to boil the water, it should be adjusted so that the flame spreads on the asbestos pad in a spot about an inch in diameter.

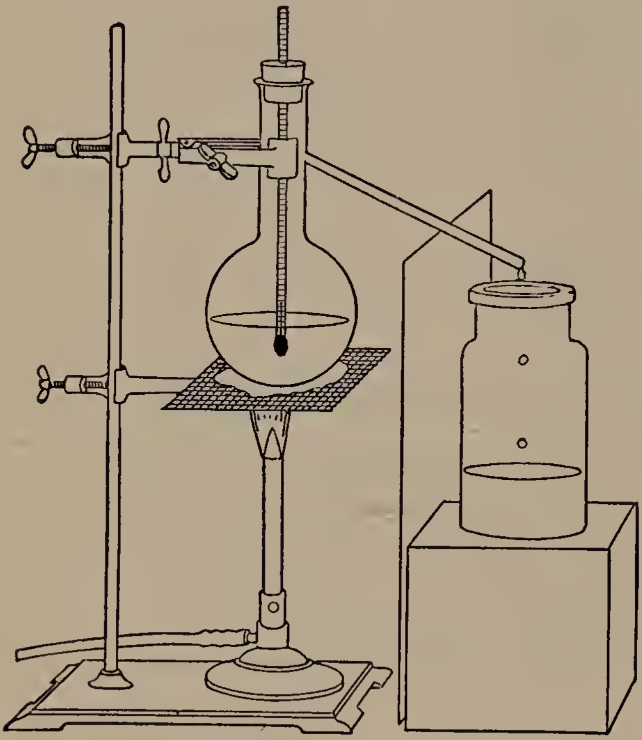

Fig. 53. Finding the boiling point of a solution.

Read and record the temperature as soon as steam issues freely from the exit-tube. The presence of some common glass beads in the flask will cause the water to boil more steadily and will prevent "bumping." Continue to heat the water to see whether the temperature rises higher.

b. Alcohol. Fill the boiling flask half full of alcohol and determine its boiling point.

CAUTION. **Alcohol vapor is inflammable.** It is well to set up a cardboard between the flask and the delivery tube in order to avoid any possibility of igniting the escaping alcohol vapor with the flame.

c. Alcohol and water. Fill the boiling flask half full of a solution of alcohol and water (equal volumes of each) and determine its boiling point. *Does it remain constant as the liquid continues to boil? Compare the boiling point of this mixture with the boiling points of water and alcohol.*

d. Salt and water. In the same way determine the boiling point of a salt solution which contains 10 g. of common salt in 100 cc. of water. Then repeat, using a solution which contains 20 g. of salt in 100 cc. of water. Compute in each case the difference between the boiling point of the solution and of water. *What relation exists between these differences and the concentrations of the solution?*

e. Sugar and water. Determine the boiling point of a sugar solution which contains 20 g. of sugar in 100 cc. of water. *Compare the boiling point of the sugar solution with that of the salt solution of the same percentage concentration.*

Compute the molecular weight of common salt ($NaCl$) and of common sugar ($C_{12}H_{22}O_{11}$). *Which contains the greater number of molecules, 20 g. of salt or 20 g. of sugar?* Does this help to explain the difference in boiling points in the salt and sugar solutions?

f. Results. *How can you tell whether a given liquid (a) is pure, (b) is a mixture of liquids, or (c) contains dissolved solids?*

Optional work. Repeat these experiments with the thermometer raised higher in the stopper so that the bulb is just below the side tube. This gives the temperature of the vapor of the boiling liquid. Compare the temperatures of the vapor of the boiling liquids with the temperatures of the liquids. Explain any differences.

EXPERIMENT 32

FREEZING POINTS OF SOLUTIONS

Apparatus. — Pint fruit jar or small battery jar. Test tubes. Thermometer.

Materials. — Cracked ice or snow. Rock salt. Salt solutions (10 g. in 100 cc. water and 20 g. in 100 cc. water). Sugar solution (20 g. in 100 cc. water).

a. Freezing mixture. Make a freezing mixture by filling a pint fruit jar, or a jar of about the same size, with a mixture of cracked ice and salt (about 3 to 1). Make a hole in the center so that a test tube may stand with its lower part surrounded by the freezing mixture (Fig. 54).

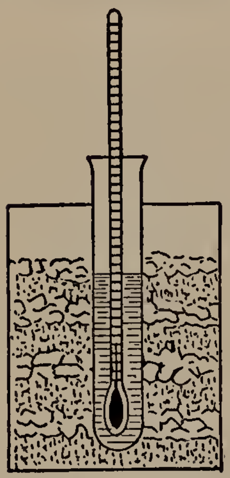

Fig. 54. Finding the freezing point of a solution.

b. Water. Pour some distilled water into the test tube (about an inch in depth), place it in position, and stir with the thermometer. From time to time raise the test tube a little and examine the contents. The ice will separate as flaky crystals. When it begins to do so, lift the tube from the freezing mixture, stir, and read the thermometer. Wait until the ice just begins to melt perceptibly; then stir and take another reading. If the two readings do not agree, find the average and call this the freezing point.

c. Solutions. In this way determine the freezing points of the following solutions:

Solution I — 10 g. common salt in 100 cc. water;
Solution II — 20 g. common salt in 100 cc. water;
Solution III — 20 g. common sugar in 100 cc. water.

d. Results. Calculate in each case the difference between the freezing point of water and that of the solution. *Does salt seem to lower or raise the freezing point of water? What is the* *effect of varying the concentration of the salt solution? Why does the sugar solution produce so much less effect on the freezing point than the salt solution?*

Optional work. Experiments show that in the case of nonelectrolytes one gram molecule of a substance dissolved in 1000 cc. of water lowers the freezing point from 0° C. to −1.86° C. Compute the gram-molecular concentration of the salt solutions used and then the depression of the freezing point as if it were not ionized at all. Finally, compute the degree of ionization, *i.e.* the per cent of the molecules which are ionized

EXPERIMENT 33

Forms of Sulfur

Apparatus. — Test tubes and rack. Crystallizing dish. Bunsen burner. Magnifying glass. Dish of water. Clamp. Funnel. Beaker.

Materials. — Roll sulfur. Carbon disulfide. Filter paper (10 cm. in diameter). Dime or other piece of silver.

a. Crystals obtained from solution. Put about 2 or 3 g. of powdered roll sulfur in a test tube, add about a quarter of a test-tubeful of carbon disulfide, and shake the tube.

Caution. **Carbon disulfide is a volatile liquid which easily takes fire. No flame should be near it.**

Pour off the clear solution (Fig. 55 A) into a crystallizing dish. Set it aside to evaporate the liquid at some distance from a flame.

(While waiting for the crystals to form, proceed with part **b**.)

When crystals have formed, examine them with a magnifying glass and note their shape; put them in a dry test tube and label them **rhombic sulfur.** *Draw several crystals in your notebook.*

b. Crystals obtained by slowly cooling molten sulfur. Place a folded filter paper in a funnel and have a dish of water at hand.

Fill a clean, dry test tube half full of powdered sulfur. Melt the sulfur very slowly by holding the test tube in an inclined position well above the flame (Fig. 55 b) and by turning the tube so as not to overheat the sulfur at any point. The liquid sulfur should be pale yellow; if it becomes dark in color, it shows that it has been overheated.

Pour the liquid sulfur into a folded filter paper (Fig. 55 c) and watch the crystals form. As soon as they extend from the sides to the center of the cone, break the crust and pour the sulfur which is still liquid into the water in the dish. At once open up the filter paper and examine the crystals with a lens. *Make a drawing of a few of them and record their color and transparency.* This form of sulfur is called **prismatic sulfur.**

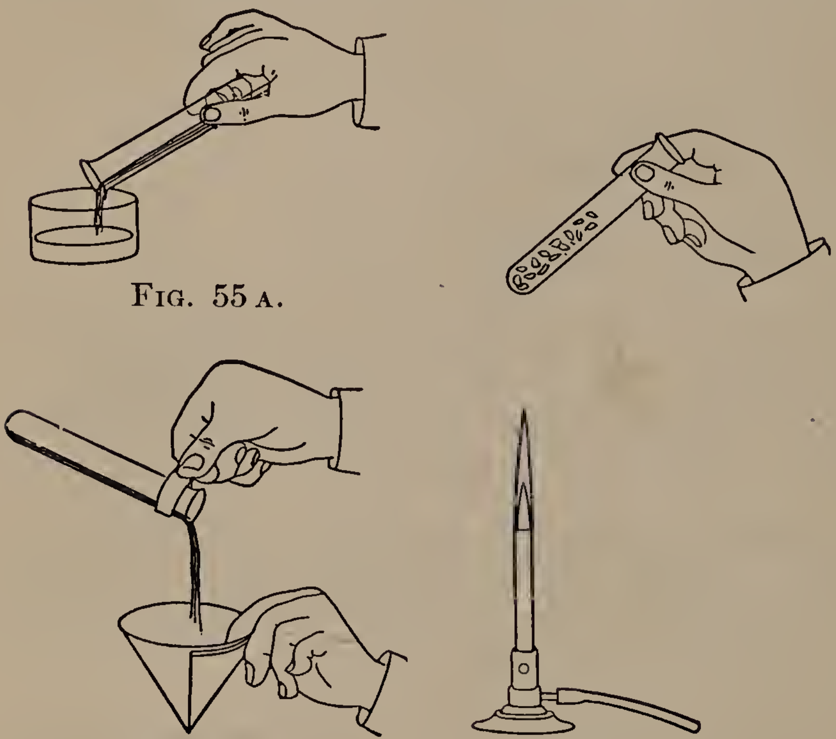

Fig. 55 a.

Fig. 55 c.

Fig. 55 b.

Fig. 55. A, Pouring a solution of sulfur into a crystallizing dish. B, Melting sulfur in a test tube. C, Pouring melted sulfur into a filter paper.

Keep some of these crystals and observe any change in their appearance.

c. Plastic sulfur, obtained by heating sulfur to its boiling point and then suddenly cooling it. Fill a beaker with water and place it on the table beside you. Half fill a test tube (preferably the one used in **b**) with small pieces of roll sulfur, hold the test tube in some kind of clamp (a folded strip of paper will serve), and very gently heat the sulfur until it melts. When it is completely melted, heat it more strongly and observe the changes in color and fluidity.

When the sulfur is boiling vigorously, pour it into the beaker of water (Fig. 56).

Caution. Do not become alarmed if the sulfur vapor begins to burn at the mouth of the test tube; do not, however, spill burning sulfur on the table.

Observe what happens to the sulfur which remains in the test tube as it slowly cools.

Take the solid from the beaker and examine it. *Note its color; is it hard or soft? is it elastic or brittle?* This form of sulfur is called **plastic sulfur** or amorphous sulfur.

Find out whether plastic sulfur is soluble or insoluble in carbon disulfide.

d. Test for free sulfur. Sulfur combines very readily with silver to form a black sulfide. Heat a minute particle of sulfur on a silver coin; a black stain is produced.

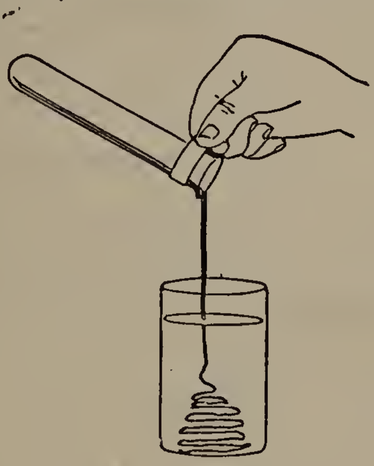

Fig. 56. Pouring boiling sulfur into cold water.

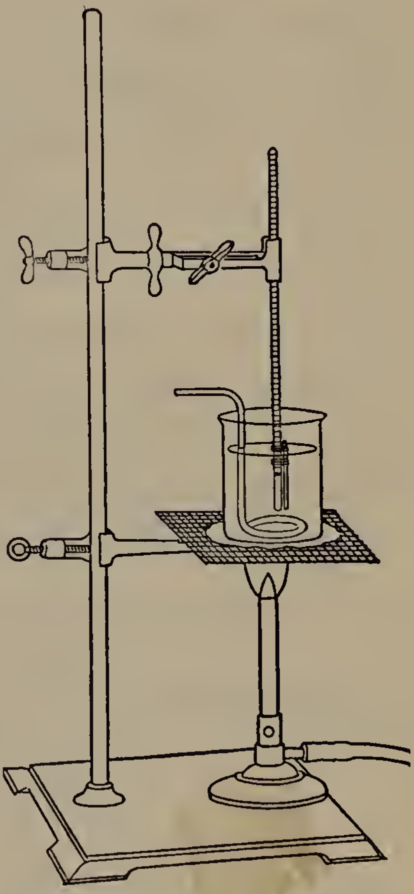

Fig. 57. Determining the melting point of sulfur.

Optional work. Devise a suitable method for determining the **densities** of roll sulfur and plastic sulfur. If your method is approved by the instructor, carry it out.

Determine the **melting points** of rhombic sulfur and prismatic sulfur. Put small fragments of the two forms of crystals into separate melting-point tubes and attach them with rubber bands to the bulb of a thermometer, as shown in figure 57. Clamp the thermometer so that its bulb dips into some olive oil in a beaker. Heat gently and stir the oil constantly until the first sign of melting occurs. *Record the temperature.*

EXPERIMENT 34

Hydrogen Sulfide

Apparatus. — Ring stand and clamp. Large test tube (20 × 2.5 cm.) and 1-hole stopper to fit. Test tubes and rack. Bunsen burner. Glass tubing. Wide-mouth bottle. Glass nozzle.

Materials. — Iron sulfide. Dilute hydrochloric acid. Solutions of lead nitrate, copper sulfate, and cadmium nitrate. Litmus papers.

a. Preparation. When iron filings and powdered sulfur are mixed together and heated, the product is iron sulfide (ferrous sulfide). Carefully slide two or three pieces of iron sulfide (FeS) into a large test tube and pour in enough dilute hydrochloric acid to cover the iron sulfide.

Note the odor of the gas. Do not inhale much of it as it is somewhat poisonous. very bad. — rotten egg

b. Tests. The odor of the gas is a sufficient test when considerable quantities of the gas are present; but a more sensitive test is to dip a strip of filter paper into a solution of lead nitrate and to hold it in the gas to be tested.

Complete the equation for preparing hydrogen sulfide (H_2S):

$$FeS + 2\,HCl \longrightarrow \underline{\;FeCl_2\;} + \underline{\;H_2S\;}\uparrow$$

Also complete the equation for testing for the gas with lead nitrate:

$$H_2S + Pb(NO_3)_2 \longrightarrow \underline{\;PbS\;}\downarrow + \underline{\qquad\qquad}$$

c. Properties. Fit the test tube with a 1-hole stopper and delivery tube as shown in figure 58. Collect the gas in a dry test tube by the upward displacement of air, remove the delivery tube, and light the gas in this test tube. As soon as the gas in the test tube burns quietly, attach a glass nozzle* or jet to the delivery tube and set fire to the gas.

---

* A glass nozzle or jet is made by heating a straight piece of glass tube in the Bunsen flame and when soft, pulling it out so that the bore is reduced. Then cut the tube off at the constricted portion.

Cautiously smell the gas around the flame. Hold a cold, dry bottle over the flame and note whether any product of the combustion is condensed therein. *What are products of the complete combustion of hydrogen sulfide? Write the equation.*

Extinguish the flame and insert the delivery tube to the bottom of a test tube which is three-fourths full of water. Let the gas bubble through the water 3 or 4 minutes. (If the action in the generator slackens, gently warm the large test tube.) *What effect has this solution on blue litmus paper?* red This solution is called **hydrosulfuric acid.**

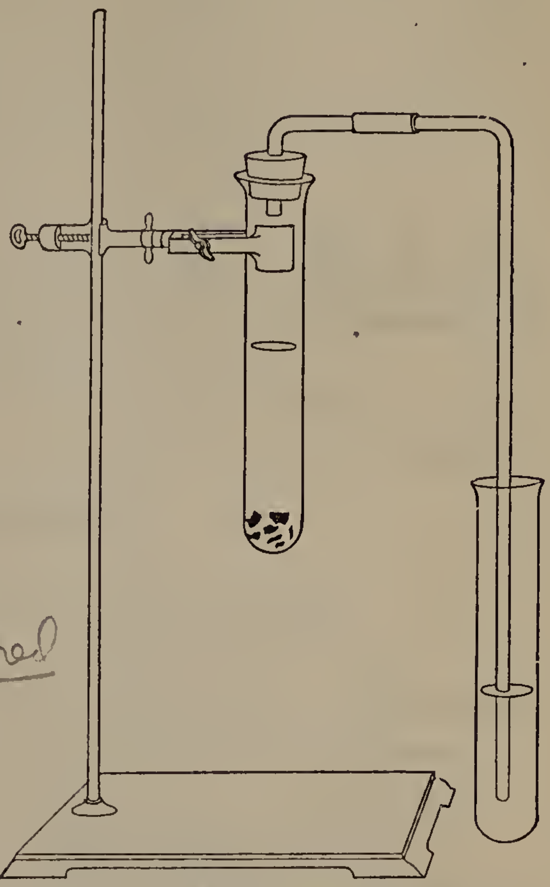

Fig. 58. Preparing a solution of hydrogen sulfide.

d. Uses. A solution of hydrogen sulfide is a very useful reagent in chemical analysis to determine what metals are present in a material of unknown composition. Some of the metallic sulfides are insoluble and have distinctive colors.

Add a portion of the hydrosulfuric acid just prepared to a solution of copper sulfate. *What are the products formed? Complete the equation:* dark.

$$H_2S + CuSO_4 \longrightarrow \underline{CuS} \downarrow + \underline{\qquad}$$

Add another portion of the hydrosulfuric acid to a solution of cadmium nitrate. *What are the products formed? Complete the equation:* yellow

$$H_2S + Cd(NO_3)_2 \longrightarrow \underline{CdS} \downarrow + \underline{2HNO_3}$$

e. ***Summarize** the observed properties of hydrogen sulfide.*

Optional work. Arrange six test tubes in a rack and fill each about one-third full of one of the following solutions: — **zinc sulfate** ($ZnSO_4$), **tin chloride** ($SnCl_2$), **magnesium chloride** ($MgCl_2$), **sodium chloride** ($NaCl$), **antimony chloride** ($SbCl_3$), and **lead nitrate** ($Pb(NO_3)_2$). Bubble hydrogen sulfide through each solution in turn. *Note whether a precipitate is formed or not.*

If no precipitate is formed, add a little ammonium hydroxide (NH_4OH) and *note any change.* If a precipitate is formed, filter, wash, dry, and heat in a bulb tube (Fig. 59) to find out whether the solid is a sulfide.

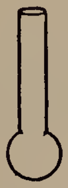

Fig. 59. Bulb tube used to heat a sulfide in.

EXPERIMENT 35

Sulfur Dioxide and Sulfurous Acid

Apparatus. — Platinum wire. Bunsen burner. Bulb tube. Ring stand with ring and clamp. Asbestos gauze. Erlenmeyer flask (250 cc.) with 2-hole stopper. Funnel. Pinchcock. Rubber connection tube. 3 wide-mouth bottles and glass squares. Test tubes. Evaporating dish. Glass tubing.

Materials. — Sulfur. Iron pyrite. Sodium bisulfite. Dilute hydrochloric acid. Pink flower. Thin slice of apple. Litmus papers. Zinc. Lead nitrate test paper.

a. Preparation. Touch a bit of sulfur with a warm platinum wire and bring the wire with adhering sulfur again into the flame. Withdraw it and observe the color of the flame of burning sulfur; also note the odor of the gas produced. *Write the equation.*

Heat (or roast) in a little bulb tube (Fig. 60) a few particles of iron pyrite (FeS_2). *What is the solid which condenses on the cool part of the tube?*

A convenient method of preparing sulfur dioxide in the laboratory is to drop hydrochloric acid slowly on sodium hydrogen sulfite ($NaHSO_3$). This reaction may be expressed by the following equation:

$$NaHSO_3 + HCl \longrightarrow NaCl + H_2O + SO_2 \uparrow$$

Arrange the apparatus as shown in figure 61. Place in the flask about 10 g. of sodium bisulfite (sodium hydrogen sulfite) and replace the stopper carrying the funnel tube and delivery tube. Attach a straight glass tube to the delivery tube in order to make it reach down to the bottom of a wide-mouth bottle.

Fill up the funnel with dilute hydrochloric acid and then adjust the pinchcock so as to admit the acid into the flask drop by drop. To increase the speed of the reaction, warm the flask *very gently* from time to time as needed.

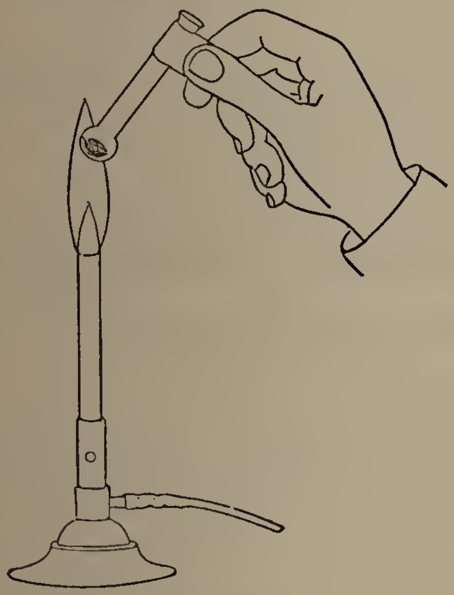

Fig. 60. Heating iron pyrite in a bulb tube.

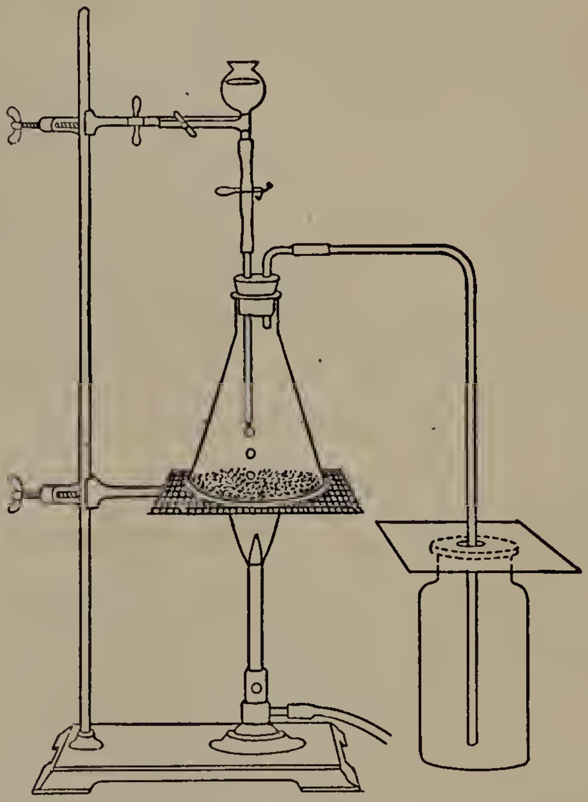

Fig. 61. Dropping hydrochloric acid on sodium bisulfite to generate sulfur dioxide.

As soon as the air in the flask has been swept out, collect two bottles of the gas by upward displacement of air. Cover each bottle with a glass plate. Boil half a test-tubeful of water so as to drive off the dissolved air and then cool under running water. Insert the delivery tube to the bottom of the boiled water and let the gas bubble through for 5 minutes. Cork this test tube just as soon as the delivery tube is taken out in order to keep out the air.

b. Properties. Uncover one of the bottles of gas just enough to pour in a little water; then close it *tightly* with the palm of the hand and shake the contents (Fig. 62). Is the hand held against the mouth of the bottle by atmospheric pressure (suction) or not? *Is the gas soluble?*

In the second bottle of gas place a moist pink flower (carnation) and a thin slice of apple. Leave another slice of apple exposed to the air outside. Compare the pieces. *What is the effect of sulfur dioxide on vegetable coloring matter?*

Fig. 62. Shaking a bottle containing sulfur dioxide and water.

c. Sulfurous acid. Test the aqueous solution made in part (a) with litmus papers. Write the equation for the formation of sulfurous acid (H_2SO_3). *Is sulfur dioxide an acidic or a basic oxide?*

Boil 5 cc. of this solution slowly in an evaporating dish and note the odor from time to time. Write the equation which represents this change and compare it with the preceding equation.

To detect sulfurous acid, we may reduce it to hydrogen sulfide by using a suitable reducing agent, such as nascent hydrogen. Put a few pieces of zinc in a test tube and cover the metal with dilute hydrochloric acid. As soon as the action becomes vigorous, test the gas with a strip of moist lead nitrate paper (the paper should remain white). Now pour in 2 or 3 cc. of sulfurous acid and test again with lead nitrate paper.

d. ***Summarize*** *the properties of sulfur dioxide: color, odor, density as compared with air, and solubility.* Also ***summarize*** *the properties of sulfurous acid: action on vegetable coloring matter, stability, oxidizing or reducing agent.*

Optional work. Sulfurous acid and its salts, the sulfites, are often used as **food preservatives,** even though their use is questionable. **To test for sulfurous acid or sulfites in foods,** apply the method of part (c) and reduce the sulfurous acid, if present, to hydrogen sulfide. Examine one or more of the following: molasses, lime juice, mushrooms, Hamburg steak, sausage, catsups, fruit juices, and dried fruits.

Put about 25 g. of the sample (macerated if a solid) into a small Erlenmeyer flask (200 cc.). Add water, if necessary, to make a thin paste. Introduce about 5 g. of sulfur-free zinc and about 15 cc. of chemically pure concentrated hydrochloric acid (HCl). Over the mouth of the flask place a small filter paper which has been moistened with a strong solution of lead nitrate ($Pb(NO_3)_2$). Warm the solution gently. Blackening of the filter paper (not merely browning) indicates the presence of sulfites.

EXPERIMENT 36

Sulfuric Acid

Apparatus. — Test tubes and rack. Bunsen burner.

Materials. — Concentrated sulfuric acid. Zinc. Dilute sulfuric acid. Sodium chloride. Wooden splint. Dilute solutions of sodium sulfate, zinc sulfate, and copper sulfate. Barium chloride solution. Dilute hydrochloric acid.

a. Heat of dilution. Fill a test tube about one-third full of water and then pour into it very gradually an equal volume of concentrated sulfuric acid. Touch the outside of the test tube and record any change in temperature. hot.

Caution. Remember that the concentrated sulfuric acid is to be poured slowly *into* the water. The reverse order produces dangerously explosive spattering.

b. Action on zinc. Place a strip of zinc in a test tube and cover it with concentrated sulfuric acid. If there is no action, warm the acid cautiously.

Caution. Hot concentrated sulfuric acid causes very severe burns when it touches the skin. Do not pour hot concentrated sulfuric acid into water. Let it cool. Slow

Place another strip of zinc in a test tube and cover it with dilute sulfuric acid. *Name the products formed in this reaction and write the equation.*

$Zn + H_2SO_4 \longrightarrow ZnSO_4 + H_2\uparrow$

Compare the action of concentrated and dilute sulfuric acid on zinc. slow. Rapid. none

c. Action on salt. Try the effect of both dilute and concentrated sulfuric acid on common salt (sodium chloride). *Identify the gaseous products.*

d. Action on wood and paper. Insert a splinter of wood into a little concentrated sulfuric acid in a test tube and let it stand for a few minutes.

Dip a glass rod into sulfuric acid and write with it on paper. Warm the paper gently over a flame. *What property of sulfuric acid do these experiments with wood and paper illustrate?*

e. Test for a sulfate. Prepare in separate test tubes dilute solutions of *sulfuric acid, sodium sulfate, zinc sulfate,* and *copper sulfate.* Add to each solution a few drops of barium chloride solution ($BaCl_2$) and then dilute hydrochloric acid. *What insoluble precipitate is formed in each case?* Barium Sulfate

To a solution of sodium phosphate add a few drops of barium chloride and dilute hydrochloric acid. *Why is it necessary to add hydrochloric acid in testing for sulfates?*

How would you distinguish between a sulfide, a sulfite, and a sulfate?

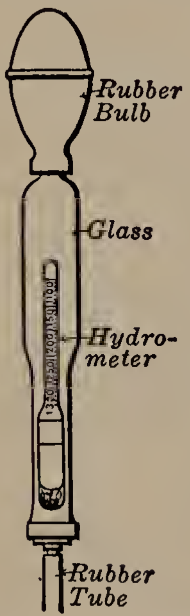

Fig. 63. Syringe hydrometer for testing a storage battery.

Optional work. One of the most important differences in the physical properties of dilute and concentrated sulfuric acid is that of density. To determine the density of an acid take a small flask (25 cc.) and paste a label around the neck so that its upper edge is exactly horizontal. Find the weight of the flask empty and weigh the flask filled with water so that the lowest part of the water surface is exactly at the upper edge of the label. Then fill the flask to the same level with dilute sulfuric acid and find the weight. Still again find the weight when filled with concentrated sulfuric acid.

Calculate the density of these acids.

A very convenient method of testing a storage battery is to determine the density of its electrolyte (dilute sulfuric acid) by means of a hydrometer (Fig. 63). Find out how this is done in a service station.

EXPERIMENT 37

AMMONIA — PREPARATION AND PROPERTIES

Apparatus. — Large test tube (20 × 2.5 cm.) fitted with 1-hole stopper. Ring stand with ring and clamp. Bunsen burner. 3 wide-mouth bottles. Glass squares. Pneumatic trough. Glass tubing.

Materials. — Slaked lime. Ammonium chloride. Litmus papers. Tapers. Concentrated hydrochloric acid.

a. Preparation. Ammonia (NH_3) can be readily produced from an ammonium salt, such as sal ammoniac (NH_4Cl), by heating it with any of the three common alkalies. Since lime is the cheapest alkali, it is the one commonly employed. As the gas, ammonia, is lighter than air, we may collect it in an inverted bottle by displacing the air.

Mix on a piece of paper 2 parts of powdered slaked lime ($Ca(OH)_2$) and 1 part of ammonium chloride (NH_4Cl). Place this mixture in a large test tube which is provided with a delivery tube, as shown in figure 64. Do not fill the tube more than half full. Clamp it, incline slightly downward in order that any condensed moisture may not run back and crack the tube.

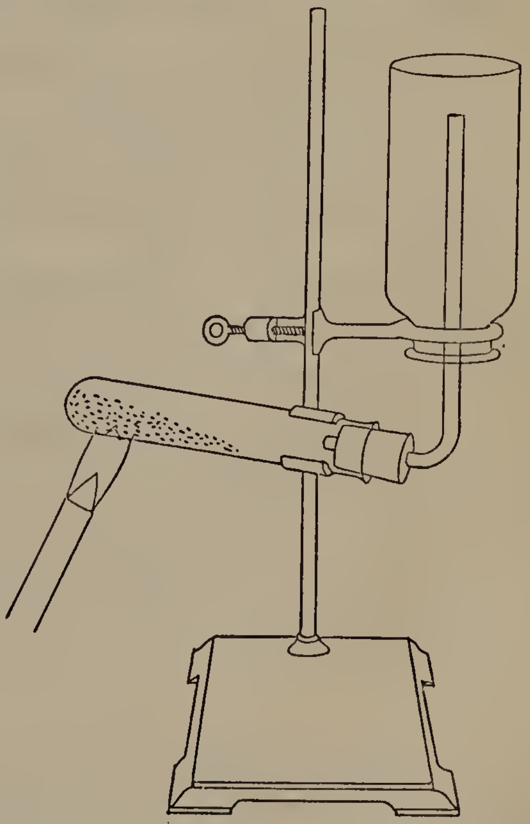

Fig. 64. Preparing ammonia and displacing the air.

Warm the mixture very gently, beginning with that portion near the mouth of the tube and gradually extending the heat to other portions, but keep the flame moving continually. As soon as the smell of ammonia shows that the gas is escaping from the bottle, lift it up and cover its mouth with a glass plate. Set it aside mouth downward. In this way collect 3 bottles of the gas.

b. Properties. Put one bottle mouth downward into a basin of water (pneumatic trough). Remove the glass plate and shake the bottle a little. *Note what happens.* Replace the glass plate and remove the bottle with its contents. Test the liquid with litmus papers. *What is the action of ammonia on water? Write the equation.*

Pour into a warm, dry bottle two or three drops of concentrated hydrochloric acid. Cover this bottle with a glass plate and invert it upon a covered bottle of ammonia. Keep the bottles mouth to mouth while you remove both plates at the same time. The white solid which is formed is ammonium chloride (NH_4Cl). *Write the equation for the reaction.*

Push a lighted taper up into an *inverted* bottle of the gas. *Does the gas burn? Does it support combustion of a taper?* Apply a lighted taper to the gas issuing from the exit-tube of the generator. *Observe whether the gas burns* (1) *while the lighted taper is present and* (2) *after it is removed.*

c. Results. Make a *list of the properties of ammonia* which you have observed in this experiment. Complete the equation for its preparation:

$$Ca(OH)_2 + 2\,NH_4Cl \longrightarrow \underline{\qquad\qquad} + \underline{\qquad\qquad} + \underline{\qquad\qquad}\uparrow$$

How could you prove that a given substance was an ammonium salt?

Optional work. To investigate other sources of ammonia, gently heat a mixture of gelatin and soda lime (sodium hydroxide and calcium hydroxide) in a test tube. Use very small equal amounts of these materials. *Note the odor of the gases given off and also their effect on moistened litmus paper.*

Smell ammonium sulfate and also slaked lime. Then rub these two substances together in the palms of the hands. *Note the odor and the effect on moistened litmus paper held over the mixture.*

Heat in a test tube a mixture of sodium hydroxide solution and ammonium nitrate. Test for ammonia with a drop of concentrated hydrochloric acid on a glass rod.

Investigate the dry "Ammo" powder. *What is its probable composition?*

EXPERIMENT 38

Analysis of Household Ammonia

Apparatus. — 10-cc. pipette. Small beaker. Ring stand and clamp. Burette.

Materials. — Bottle of "household ammonia." Methyl-orange solution. Normal hydrochloric acid solution.

a. Preliminary. The value of "household ammonia" depends upon the amount of base (NH_4OH) which it contains. It often happens that some brands contain one-tenth as much as others and yet are retailed at the same price. There is no way in which the average consumer can detect the difference and often the grocer does not know the concentration of the brand he sells. However, it is quite easy for a student of chemistry to measure the concentration of a sample of household ammonia by titration with an acid of known concentration. Our first step, then, is to review the experiment on the neutralization of a base and an acid by titration (Exp. 20).

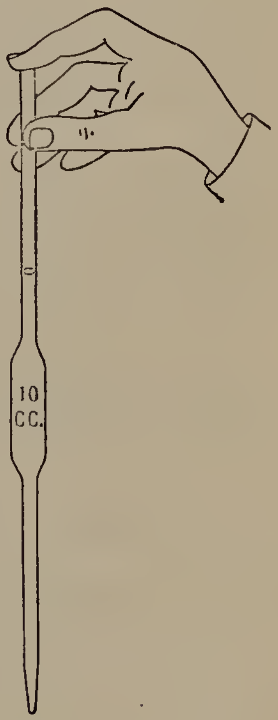

Fig. 65. A 10-cc. pipette.

b. Procedure. Bring from home or buy of a grocer at least 100 cc. (4 oz.) of household ammonia. Take a 10-cc. pipette (Fig. 65) and rinse it out with the ammonia solution (throw away the rinsings). Measure out very carefully 10 cc. of the household ammonia with a pipette as follows: Insert the jet end of the pipette deeply in the liquid and place the other end in the mouth. By drawing (sucking) the air out of the pipette, the liquid is forced up into it. As soon as the liquid rises above the 10-cc. mark on the stem, remove the pipette from the mouth and quickly cover the end with the finger. By moving the finger slightly, the liquid is allowed to flow out until the surface is just on the mark.

Let this carefully measured volume of household ammonia flow out into a clean beaker and add about 20 cc. of distilled water. Add two or three drops of methyl-orange solution to the mixture. This indicator turns red with acids and yellow with bases.

Fill a burette with a normal solution of hydrochloric acid. Take the usual precautions about rinsing out the burette with a little of the solution to be used in it and see that the tip is filled with the solution. *Read and record the surface of the acid solution* (which must be on the graduated portion of the burette). Then place the beaker on a sheet of white paper directly under the burette and let the acid flow slowly into the ammonia solution, stirring constantly, until the change of color shows that enough acid has been added. In fact, too much acid has doubtless been used in this preliminary trial, but *note the volume of acid actually used.*

In the next trial the acid may be drawn off rapidly until the volume used has come within 2 cc. of that employed in the first trial. Now proceed very slowly until a single drop changes the color of the indicator. Make several trials until three consecutive trials agree within 0.5 cc. Find the average of these three consistent values for the volume of normal hydrochloric acid which neutralizes 10 cc. of the household ammonia.

c. Results. Compute the volume of normal acid required to neutralize 1 cc. of the ammonia solution. This volume, or **factor**, expresses the multiple of normality of the ammonia solution. If this volume is less than one, it expresses the fractional normality of the ammonia solution. A normal ammonia solution contains 17 g. of ammonia per liter, or is about 1.7 per cent ammonia. Therefore the product of the normality of the ammonia solution and 1.7 gives the per cent of ammonia in the household ammonia.

Arrange the data and results in tabular form and show each step in the computation.

Optional work. The vital question with the consumer is **how much ammonia do I get for my money?** To answer this question we must know not only the concentration of the solution but also the volume of the container (bottle) and its cost. Purchase two bottles of different brands of household ammonia, and determine the concentration of each and the volume of the bottles. Calculate in each case the amount (g.) of ammonia obtained for one dollar.

EXPERIMENT 39

NITRIC ACID

Apparatus. — Tubulated retort (250 cc.). Tripod. Bunsen burner. Asbestos gauze. Ring stand and clamp. Large test tube. Pneumatic trough. Funnel. Test tubes.

Materials. — Sodium nitrate. Concentrated sulfuric acid. Excelsior. White woolen yarn. Concentrated ammonium hydroxide. Copper turnings. Zinc. Ferrous sulfate solution, freshly prepared.

CAUTION. Nitric acid is very corrosive. Do not drop it on your flesh or clothes.

a. Preparation. Place about 30 g. of sodium nitrate (or potassium nitrate) on a piece of paper and fold the paper so that the solid may be slipped into the retort through the tubulure. Support the retort on the wire gauze with asbestos center, which is itself supported on a tripod. Clamp the neck of the retort in such a way (Fig. 66) that it extends into a large test tube or small flask partly immersed in cold water. Pour 20 cc. of concentrated sulfuric acid through a funnel upon the nitrate and then carefully remove the funnel without dropping acid from it. Replace the glass stopper in the tubulure.

As soon as the sulfuric acid has moistened the entire mass of nitrate crystals, begin heating the retort gently. Distill at as low a temperature as possible until no more nitric acid condenses in the neck of the retort.

Allow the retort to cool in position. The residue, which is sodium (or potassium) hydrogen sulfate ($NaHSO_4$), may be removed with warm water. *Write the equation for the preparation of nitric acid.*

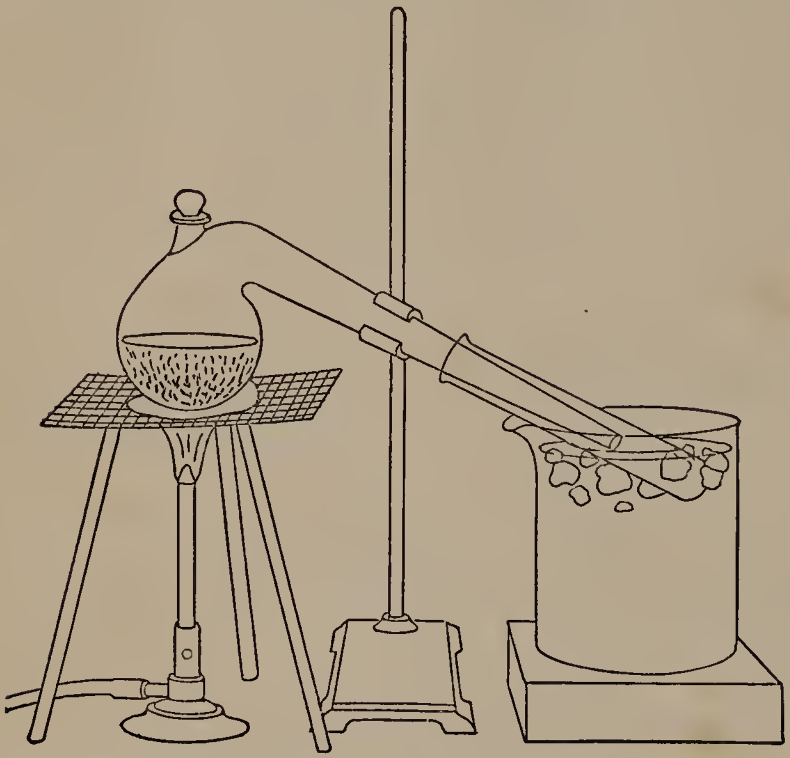

Fig. 66. Making nitric acid with a tubulated retort.

b. Properties. The nitric acid just prepared is 100% pure, and great care must be used in handling it. Pour 1–2 cc. of this acid into a test tube and insert a small, loose wad of excelsior so that it remains about an inch above the acid. Holding the test tube in a holder, boil the acid vigorously until the vapor reaches the excelsior. Now move the test tube so as to heat the excelsior itself for a few seconds. *How does this experiment illustrate the oxidizing action of nitric acid?*

Pour 1–2 cc. of the acid on a piece of white woolen yarn in a test tube, rinse with water, and then add ammonium hydroxide. This action of nitric acid followed by ammonium hydroxide serves as a **test** for those nitrogenous substances known as **proteins.**

Pour 1–2 cc. of the acid on a small piece of copper in a test tube. This action is characteristic of nitric acid. Repeat this experiment using zinc instead of copper. *Compare the action of nitric acid on metals with that of hydrochloric and sulfuric acids.*

c. Test for the nitrate (NO_3^-) ion. This is a very delicate test, and the directions must be followed with care. Pour into a test tube about 3 cc. of ferrous sulfate ($FeSO_4$) solution. Add to it 2 drops of dilute nitric acid and shake the test tube. Holding the tube in an inclined position, pour 2 cc. of concentrated sulfuric acid slowly down the side of the tube (Fig. 67). The heavy acid will run down the tube and form a separate layer under the mixture of the other two solutions. *Describe the coloration in the ring where the two layers meet.* Repeat this test using a very small amount of sodium nitrate dissolved in 2 cc. of water instead of the dilute nitric acid.

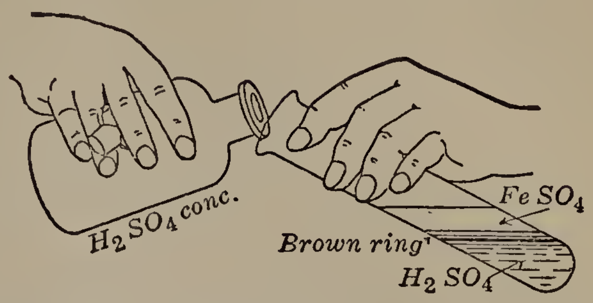

Fig. 67. Testing for nitrate ions with ferrous sulfate solution.

d. ***Summarize** the properties of nitric acid.*

Optional work. Investigate and compare the action of dilute nitric acid (1 to 10) and concentrated nitric acid on copper, magnesium, and tin.

EXPERIMENT 40

NITRIC OXIDE

Apparatus. — Wide-mouth bottle with 2-hole stopper. Thistle tube. Pneumatic trough. 3 wide-mouth bottles and glass squares. Deflagrating spoon. Large test tube with 2-hole stopper. Dropper. Glass tubing.

Materials. — Copper turnings. Concentrated nitric acid. Tapers. Asbestos paper. Red phosphorus. Sodium peroxide.

a. Preparation. Place some pieces of copper (about 10 g.) in a generating bottle and fit it with a 2-hole stopper carrying a thistle tube and a delivery tube, as shown in figure 68. Prepare to collect 3 bottles of gas over water. Pour in enough water to cover the copper and the lower end of the thistle tube and then add about half as much concentrated nitric acid. The action may be a bit slow in starting; if necessary add from time to time small quantities of the concentrated nitric acid. If the action becomes very vigorous, add cold water.

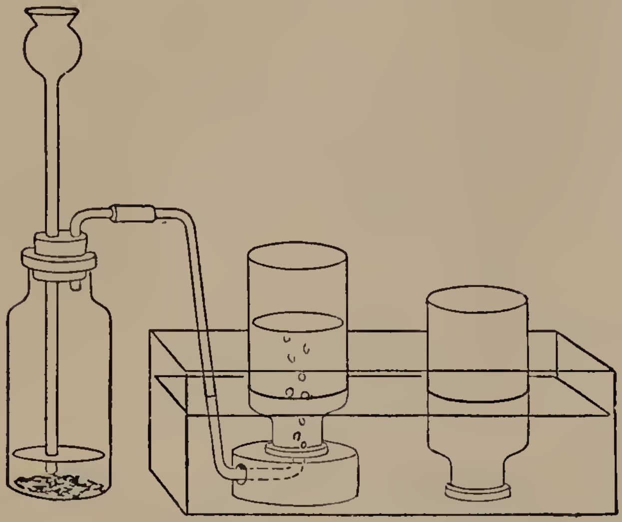

Fig. 68. Making nitric oxide.

After the air in the generator has been swept out, fill two bottles with the gas, nitric oxide (NO), and fill the third bottle half full. Let them stand in the trough for later use.

Note the color of the gas in the generating bottle just after the action starts and again later. Also observe the color of the liquid left in the generator. This color is characteristic of the cupric ion (Cu^{++}).

b. Properties. Let one bottle of nitric oxide escape into the air. *What evidence of chemical change do you observe?*

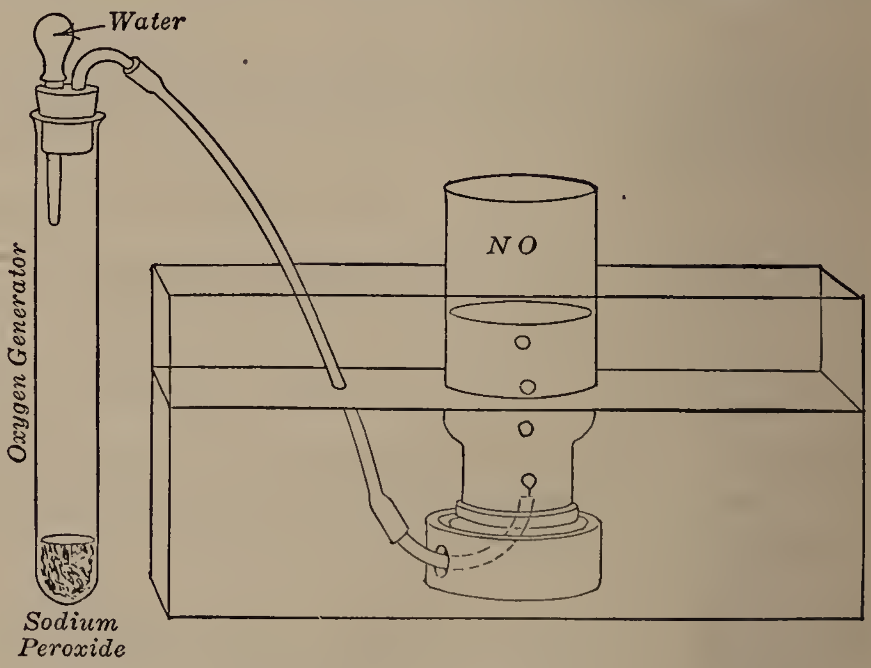

Fig. 69. Generating oxygen and introducing the gas into nitric oxide.

Cover a second bottle of the gas with a glass plate and set it upright on the table. Plunge a lighted taper into the gas, withdraw it at once and replace the plate. Line a deflagrating spoon with asbestos paper and put into it a bit of red phosphorus. Ignite the phosphorus and when it is burning vigorously plunge it into the same bottle. *Does nitric oxide support the combustion of a taper and phosphorus?*

To study the effect of oxygen on nitric oxide, set up a little oxygen generator, as shown in figure 69. Place in the large test tube about 1–2 cc. of sodium peroxide (Na_2O_2) and fill the dropper with water. Immerse the delivery tube in the trough and let the water drop a little at a time on the peroxide. When the air has been displaced from the test tube, allow the oxygen to ascend slowly into the bottle which is only half full of nitric oxide. *Note the change in color. Does the volume of the gas change? If so, in which direction? Compare the result with that obtained by allowing air to mix with nitric oxide. With what gas in the air does the nitric oxide combine to become brown?* The brown gas is nitrogen dioxide (NO_2).

c. Summarize *in parallel columns the properties of nitric oxide and nitrogen dioxide.*

Optional work. Obtain crystals from the liquid left in the generator and find the action of heat on them. Identify the substances produced.

EXPERIMENT 41

NITROUS OXIDE

Apparatus. — Small flask with 1-hole stopper. Large test tube with 2-hole stopper. Ring stand and clamp. Pneumatic trough. 4 wide-mouth bottles and glass squares. Test tube with 1-hole stopper. Glass tubing.

Materials. — Ammonium nitrate. Wooden splint. Copper turnings. Concentrated nitric acid. Anhydrous copper sulfate.

a. Preparation. Set up the apparatus as shown in figure 70. Put about 20 g. of ammonium nitrate (NH_4NO_3) into the flask and clamp it with the neck inclined slightly downward. The ammonium nitrate on heating decomposes into water and a gas, nitrous oxide (N_2O). Hence the condensing trap is inserted in the delivery tube. It is better to use warm water in the bottles and trough when collecting this gas.

The heat must be carefully regulated or an explosion may occur. Therefore it is advisable that two pupils work together on this experiment and that one of them give his *entire* attention to heating the flask gently and steadily.

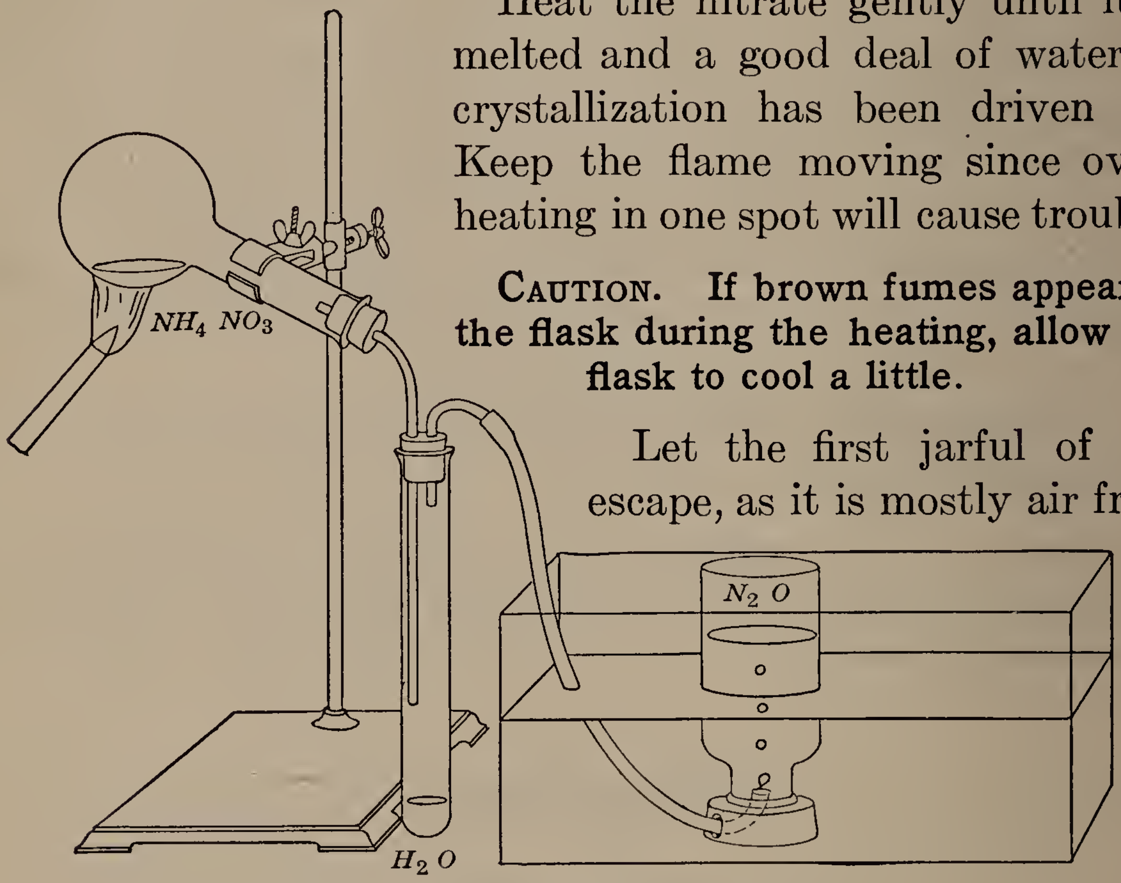

Fig. 70. Generating nitrous oxide.

Heat the nitrate gently until it is melted and a good deal of water of crystallization has been driven off. Keep the flame moving since overheating in one spot will cause trouble.

Caution. If brown fumes appear in the flask during the heating, allow the flask to cool a little.

Let the first jarful of gas escape, as it is mostly air from the flask and test tube. Collect three and a half bottles of the gas and then remove the delivery tube from the water before the heating is discontinued.

Identify the liquid in the large test tube by adding a little anhydrous copper sulfate. *Complete the equation:*

$$NH_4NO_3 \longrightarrow N_2O \uparrow + \underline{\qquad\qquad}$$

b. Properties. Describe the color and odor of the gas.

Pour a little cold water into a bottle of the gas and cover it tightly with the palm of the hand. Shake the contents of the bottle vigorously. *What does this experiment show about the solubility of the gas in cold water?*

Plunge a glowing splinter into another bottle of the gas. *What other gas have we already studied which behaves in the same way?*

Prepare some nitric oxide by putting a few pieces of copper turnings into a test tube which is fitted with a stopper and delivery tube. Cover the copper with water, add an equal volume of concentrated nitric acid, and at once insert the stopper. Let the nitric oxide bubble into water until the colored gas in the test-tube generator has disappeared. Then place the end of the delivery tube under the bottle which is half full of nitrous oxide.

Does the nitrous oxide react on nitric oxide in the same way that we found oxygen did in experiment 40*?*

c. Questions. (1) *Why is it better to collect nitrous oxide over warm water?* (2) *Why does nitrous oxide support the combustion of a burning splint while nitric oxide does not?* (3) *How would you distinguish between nitric oxide and nitrous oxide? between nitrous oxide and oxygen?*

Optional work. Investigate the action of (*a*) feebly burning sulfur and (*b*) strongly burning sulfur in nitrous oxide. Explain the difference in action. Is the same compound of sulfur formed when sulfur burns in nitrous oxide that is formed when sulfur burns in air? Give a reason for your answer.

EXPERIMENT 42

Law of Concentration or Mass Action

Apparatus. — Small flask with 1-hole stopper. Pneumatic trough. 2 wide-mouth bottles. Glass tubing. Clock or watch with second hand.

Materials. — Strips of sheet zinc (1 × 10 cm.). Dilute and concentrated hydrochloric acid. Copper sulfate solution.

a. Outline. To show the relation between the time of a reaction and the concentration of one of the reacting substances, we shall study the reaction between zinc and hydrochloric acid. If we select two similar pieces of zinc and use acids of different but known concentration, we may determine in each case the length of time required to collect a definite volume of hydrogen.

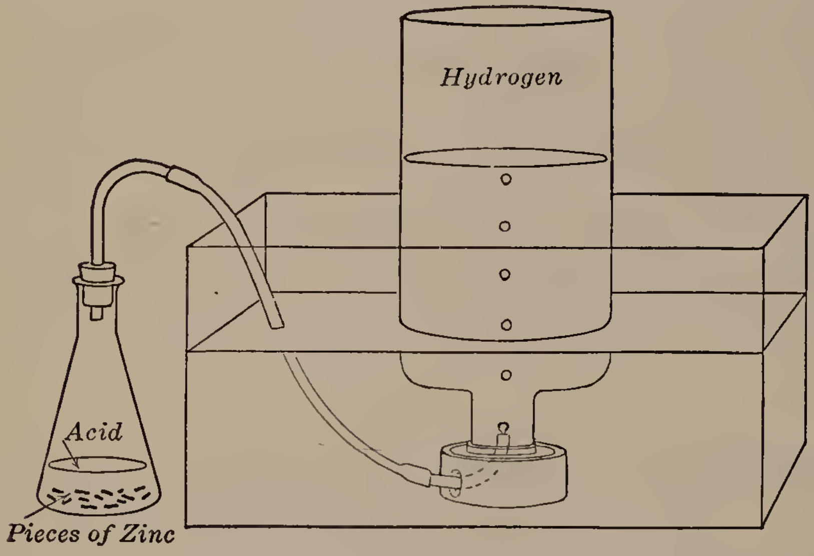

Fig. 71. Generating hydrogen with acids of varying concentrations.

b. Procedure. Cut out two strips of sheet zinc each of which is about 1 cm. by 10 cm. Clean them with dilute hydrochloric acid and then wash them thoroughly with water. To furnish a catalyst for the reaction, dip them for about half a minute in very dilute copper sulfate solution. Coil up each piece loosely.

Arrange a small flask with a 1-hole stopper and delivery tube for collecting hydrogen in a bottle over water as shown in figure 71.

c. Pour into the flask 10 cc. of concentrated hydrochloric acid and 30 cc. of water. Shake up the mixture and then slide in a zinc strip. Determine just how long it takes to collect a bottle full of hydrogen. It is well to keep the flask cool by setting it in a dish of cold water.

Repeat the experiment, using 5 cc. of concentrated hydrochloric acid, 35 cc. of water, and the second strip of zinc.

d. Questions. *How do the two pieces of zinc compare in area? What is the relation between the two acid solutions? What is the relation between the length of time required to collect a bottle full of hydrogen in the two cases?*

What is the relation between the concentration of the acid and the speed of reaction?

EXPERIMENT 43

INCOMPLETE REACTIONS

Apparatus. — Test tubes and rack. Bunsen burner. Funnel. Glass rod.

Materials. — Fused calcium chloride. Oxalic acid. Filter papers. Concentrated hydrochloric acid. Dilute ammonium hydroxide.

a. Preliminary. A double decomposition reaction will go to an end, *i.e.* is complete, (1) if one of the products is *volatile,* (2) if one of the products is *insoluble,* and (3) if one of the products *does not ionize.* There are many reactions which do not go quite to an end inasmuch as *no product is absolutely insoluble.* Let us study the reaction between calcium chloride ($CaCl_2$) and oxalic acid ($H_2C_2O_4$). This is a case of double decomposition, and the products are calcium oxalate (CaC_2O_4) and hydrochloric acid (HCl). The completeness of this reaction depends on how insoluble calcium oxalate is in hydrochloric acid.

b. Calcium chloride and oxalic acid. Dissolve 1 g. of fused calcium chloride in 10 cc. of water and dissolve 1.2 g. of oxalic acid in 10 cc. of water. Heat the solutions to the boiling point and then add the acid solution to the calcium chloride solution a little at a time. Allow the precipitate to settle between the additions. As soon as one drop of the acid solution gives no further precipitation in the clear liquid, *record* (approximately) *the fractional part of the acid which remains unused.* Then add the rest of the acid to make certain that there is more than enough to precipitate all the calcium as oxalate.

Write the equation for this reaction and write the names below the formula of each substance. Place a downward arrow (↓) *after the precipitate.*

c. Reversible reaction. To find out the action of hydrochloric acid on calcium oxalate, pour the mixture of precipitate and liquid prepared in (**b**) through a filter paper. Collect the filtrate in a test tube, label it, and set it aside. Wash the precipitate on the filter using only a little water at a time. When the filter has drained, put a test tube under the funnel, puncture the filter paper with a glass rod, and wash the precipitate down through the paper into the test tube.

To the calcium oxalate suspended in water add concentrated hydrochloric acid. *Does the precipitate dissolve or react with the acid?* If it does react, then this reaction is reversible and the acid must have acted on the calcium oxalate in the original mixture. Thus the complete precipitation of the calcium was prevented.

d. To complete the reaction. In order to precipitate the calcium oxalate completely we shall remove (that is, neutralize) the hydrochloric acid. To the solution in the test tube marked "Filtrate" add ammonium hydroxide, a drop at a time, and shake the solution. *Explain the results.*

What would be the effect of adding hydrochloric acid to the solution in (**d**)?

State the law which is illustrated in this experiment.

EXPERIMENT 44

DESTRUCTIVE DISTILLATION OF COAL

Apparatus. — Ring stand with clamp. Ignition tube (hard-glass test tube) with 1-hole stopper. Large test tube with 2-hole stopper. Pneumatic trough. Wide-mouth bottle. Bunsen burner with wing-top. Glass tubing.

Materials. — Small pieces of bituminous (soft) coal. Litmus papers. Lead nitrate test papers.

a. Distillation of coal. Fill an ignition tube about three-fourths full of small pieces of soft coal and clamp it horizontally as shown in figure 72. Have the delivery tube extend nearly to the bottom of the condensing tube (large test tube) and arrange to collect the gaseous products over water. Heat the ignition tube gently at first and then strongly until no more gas is given off. During the heating, test the stream of gas

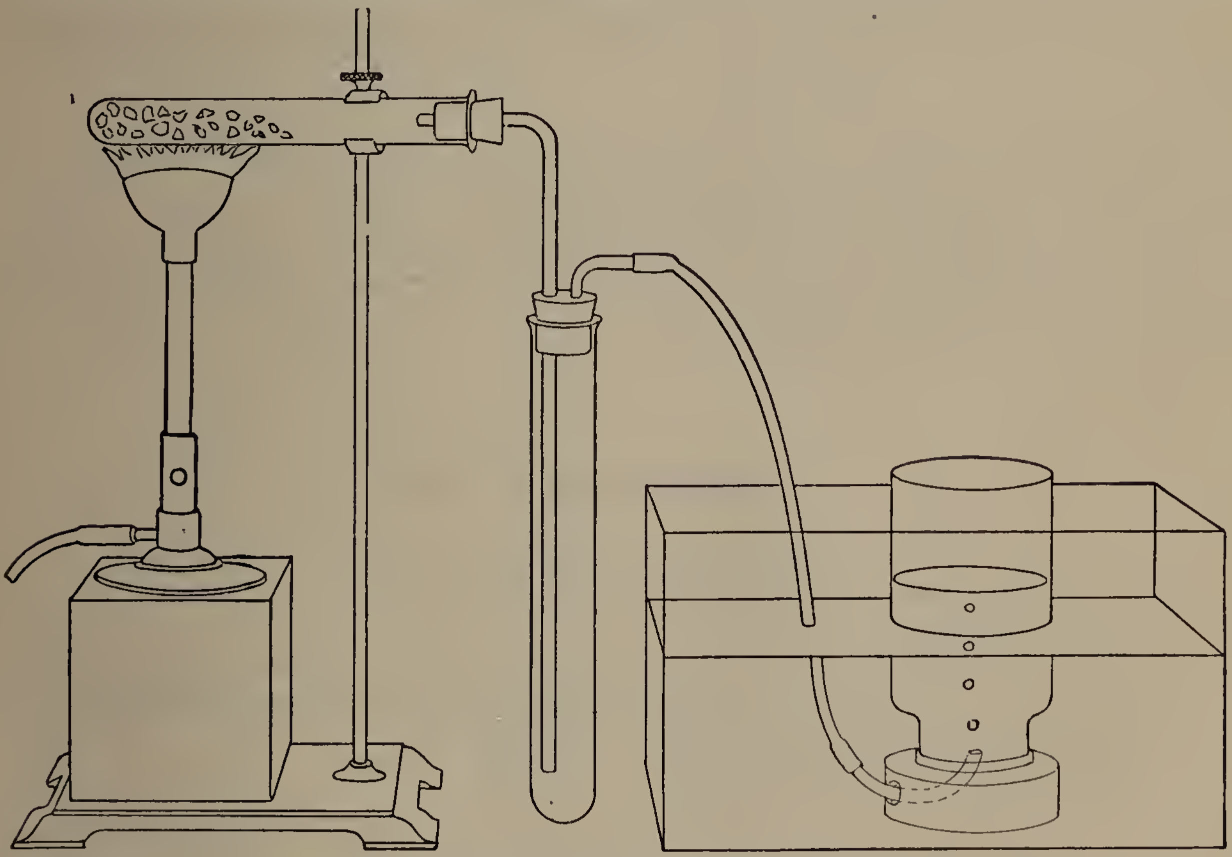

Fig. 72. Destructive distillation of coal.

in the delivery tube for hydrogen sulfide (H_2S) with a strip of paper moistened in lead nitrate solution, and also for ammonia with moistened red litmus paper. *Results?*

b. Products. Examine the gas which has been collected over water. *Does it burn?*

Observe the properties of the material in the condensing tube. *Name it.*

When the ignition tube has become cool enough to handle, shake its contents out on the base of the ring stand. *Compare this residue in color, structure, and weight with the original coal. Name it.*

Why is this process called destructive distillation?

Name three important products of the destructive distillation of coal. Name a by-product.

Optional work. In the same way carry out the destructive distillation of **wood**, using small splinters or chips. In this process the tarry distillate in the condensing tube is a mixture (called pyroligneous acid) which contains wood alcohol, acetic acid, acetone, tar, and other substances.

EXPERIMENT 45

Analysis of Coal

Apparatus. — Ring stand with ring. Pipe-stem triangle. Porcelain crucible with cover. Bunsen burner. Horn pan balance and weights. Mortar and pestle. Drying oven.

Materials. — Samples of coal (from home).

a. "Proximate analysis." When coal burns, the most evident constituents are (1) moisture, (2) volatile matter, (3) fixed carbon, and finally (4) a residue of ash. The fixed carbon is volatilized only when heated in contact with air. These several constituents cannot be sharply separated, and hence such an analysis depends upon more or less arbitrarily standardized methods. This analysis does, however, furnish an acceptable indicator of the type of coal. Coals are classified according to the relative proportion of volatile matter to fixed carbon. The ratio of fixed carbon to volatile matter is called the "fuel ratio." Thus the fuel ratio of anthracite coal is not less than 10; while the fuel ratio of bituminous coals varies usually from 3 to 6.

b. Moisture. Pulverize several pieces of coal to particles about the size of a pea and mix thoroughly. Take about a teaspoonful of this pulverized coal and grind it in a mortar to a very fine powder. Weigh to a centigram (0.01 g.) a clean, dry crucible and cover, and weigh out about 2 g. of the powdered coal. Record all these weights in tabular form:

Wt. of crucible and cover	g.
Wt. of crucible, cover and coal	g.
Wt. of coal	g.

Heat the crucible (uncovered) in an oven for an hour at 104°–107° C. Then cool and again weigh the crucible (including the cover). The loss of weight equals the weight of the moisture driven off. *Calculate the per cent of moisture in the coal.*

c. Volatile matter. Heat the covered crucible and contents from (b) on a pipestem triangle with the Bunsen flame until no more smoke comes off. Remember that a crucible should not be heated or cooled too suddenly, but when once hot it should be heated until the entire crucible is red-hot. Let the crucible cool with the cover on and then reweigh. The loss in weight during this

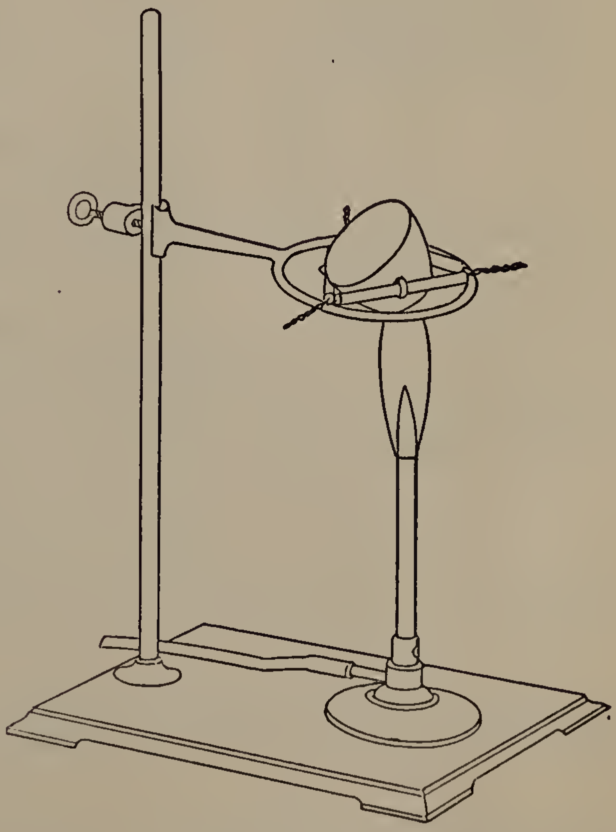

Fig. 73. Determining the ash in coal.

heating is due to the volatile matter which has been expelled. *Compute the per cent of volatile matter in the coal.*

d. Fixed carbon (coke). Remove the cover from the crucible and incline it on the pipestem triangle (Fig. 73). Heat the crucible and contents from (c) to a full red heat until all the carbon has burned off. In the same way burn off any deposit on the cover. Cool and weigh the crucible (with cover) and its residue of ash. The loss of weight during this heating is the weight of the fixed carbon. *Calculate the per cent of fixed carbon in coal.*

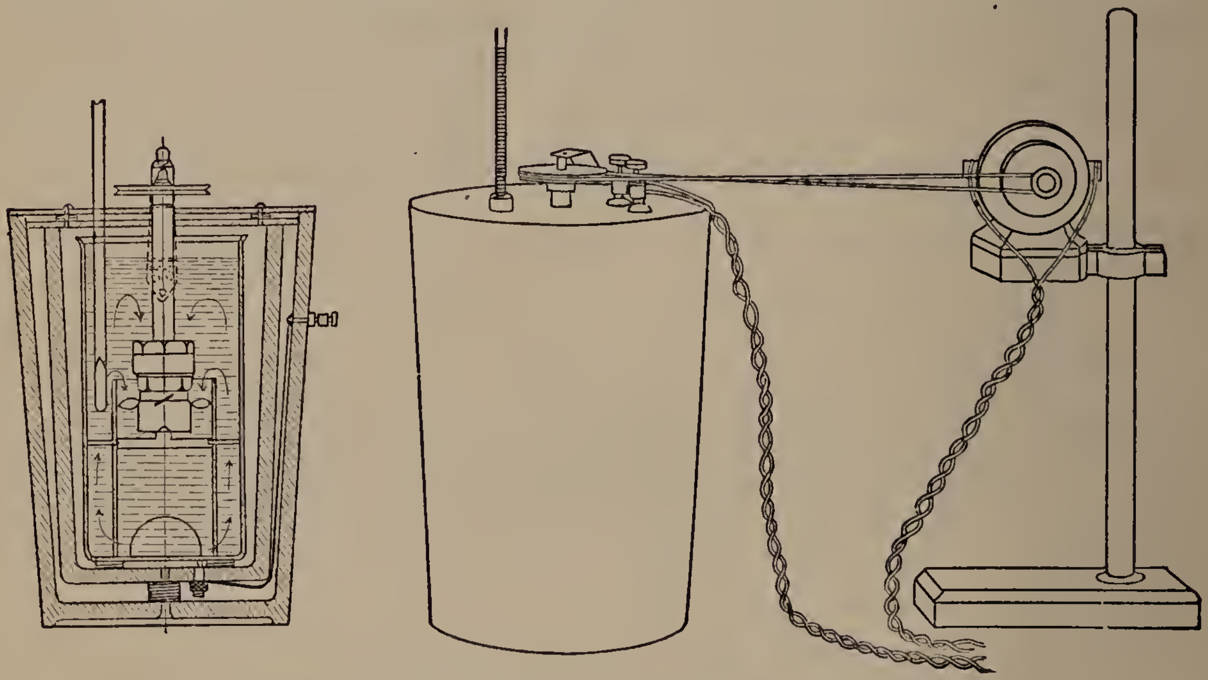

Fig. 74. Parr's peroxide bomb calorimeter.

e. Ash. The residue left in the crucible from (d) is the non-volatile mineral matter of the coal and is called ash. *Calculate the per cent of ash in coal.*

f. Compare the proximate analysis of your sample of coal with the analysis given in Engineering Handbooks (*Marks*' Mechanical Engineers' Handbook).

Optional work. If a Parr Peroxide Bomb Calorimeter (Fig. 74) is available, determine the heating value of a sample of coal, *i.e.* the B. t. u. per pound. The manipulation is simple, and the results are accurate to about 1% or less. Full directions for the manipulation and calculation are furnished with the instrument.

EXPERIMENT 46

Charcoal — Preparation and Properties

Apparatus. — Tripod. Bunsen burner. Sand crucible. Iron crucible and cover. Pipe-stem triangle. Ignition tube with 1-hole stopper. Ring stand and clamp. Test tubes. Mortar and pestle.

Materials. — Blocks of wood. Solution of molasses, brown sugar, cochineal, or indigo. Animal charcoal (boneblack). Copper sulfate solution. Hydrogen sulfide solution. Powdered copper oxide. Limewater.

a. Preparation. When animal or vegetable matter is heated strongly in the absence of air, the residue is charcoal. We may easily prepare wood charcoal by placing on a thin layer of sand in a sand crucible or on a small iron pan a few chips or small blocks of wood. If we then cover them with sand to keep out the air (Fig. 75) and heat strongly until all smoking ceases, we find, when the crucible is cool, that the residue is charcoal.

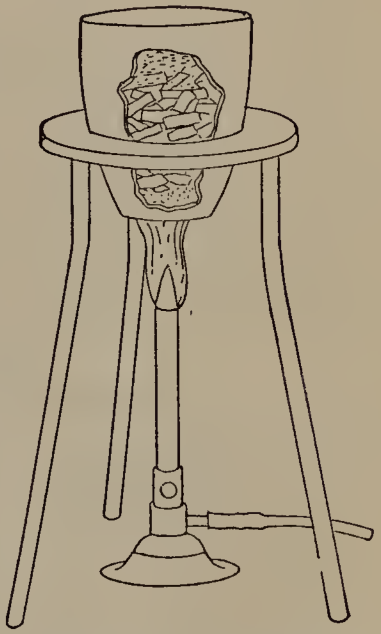

Fig. 75. Preparation of charcoal in a sand crucible.

b. Properties. (1) To show that carbon is a decolorizer, fill a test tube half full of water which has in it enough molasses, brown sugar, cochineal, or indigo to give a decided color. Add about 5 cc. of animal charcoal to the solution, shake thoroughly, and boil for 3–4 minutes. Filter the solution, and, if the filtrate is not clear and transparent, repeat the operation with fresh animal charcoal. The activity of the charcoal is much increased by previous heating in a covered iron crucible. If a solution of sugar or molasses was used, taste the filtrate to determine whether or not the sugar has been removed.

Repeat this experiment with a solution of copper sulfate.

Can the color be removed from all liquids by filtering them through animal charcoal (boneblack)?

(2) To show that carbon is a deodorizer, fill a test tube about one-fourth full of powdered wood charcoal and add about 5 cc. of hydrogen sulfide (H_2S) solution. Cork the test tube and at short intervals for 15 minutes shake the contents vigorously. Compare the odor with that of the original solution and, if it is not perceptibly removed, add more charcoal and continue shaking the contents. *What use of this property of charcoal was made in the World War?*

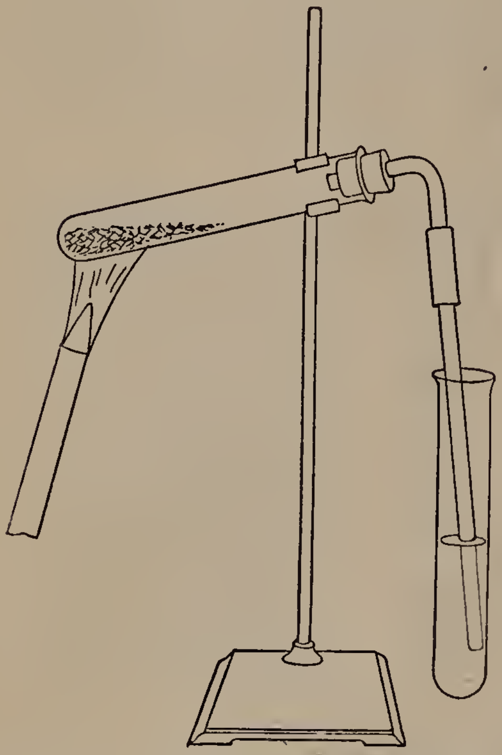

Fig. 76. Reducing copper oxide with carbon.

c. Uses. The most important chemical use of charcoal (or coke) is as a reducing agent. Mix intimately about 5 g. of copper oxide (CuO) with 1 g. of powdered wood charcoal in a mortar. By means of a folded strip of paper slide the mixture into a hard-glass test tube (ignition tube) and fit the tube with a 1-hole stopper carrying a delivery tube. Arrange the apparatus as shown in figure 76 so that the gas evolved when the tube is heated will bubble through a little limewater in a test tube.

Heat the mixture in the ignition tube at first gently to avoid cracking the tube and then as strongly as possible, beginning at the part near the stopper and gradually moving the flame toward the closed end of the tube. The tube must be heated persistently for at least 10 minutes. *What is the first change that takes place in the limewater?*

Remove the delivery tube from the limewater and stop heating. When the tube is cold pour its contents into a mortar and vigorously grind the residue under water. Let a stream of water wash away the lighter particles. *What substance is carried away by the water? What is left in the mortar? What element is taken from the copper oxide? Describe the process and write the equation of the reaction.*

d. Results. *Summarize these properties of charcoal (carbon) which you have observed in this experiment.*

Optional work. We may also use carbon to reduce such metallic oxides as tin oxide (SnO_2) and lead oxide (PbO). This same process may be utilized **to detect carbon in a compound.** Find out whether sugar, wax, and naphthalene (moth balls) contain carbon. *How could you utilize this same process to detect hydrogen in the compounds?*

EXPERIMENT 47

GASOLENE AND KEROSENE

Apparatus. — Hydrometer, Baumé scale for light liquids. Hydrometer jar or cylinder. Tripod. Bunsen burner. Asbestos gauze. Beaker. Watch glass. Wide-mouth bottle with cork. Iron dish with sand. Ring stand. Thermometer. Evaporating dish.

Materials. — Gasolene (petrol). Kerosene. Splint.

a. Specific Gravity. Although this test is not very important in determining the properties of gasolene and kerosene, yet it is worth while applying since it is an easy method of detecting differences in the commercial materials. The regular commercial hydrometer (Fig. 77) may be used to determine the specific gravity. Since the scale of the instrument is, as a rule, made for 60° F. (15.6° C.), it is usually easier to get the liquid at this temperature than to make the necessary corrections. After the specific gravity has been approximately determined, raise the hydrometer nearly down to the bulb, and wipe the stem clean; then carefully allow it to sink in the liquid. This must be done in such way that the stem of the hydrometer is not wet above the point corresponding to the specific gravity of the liquid. Now read the mark on the stem corresponding to the level of the liquid. In this way determine the specific gravity of gasolene and kerosene.

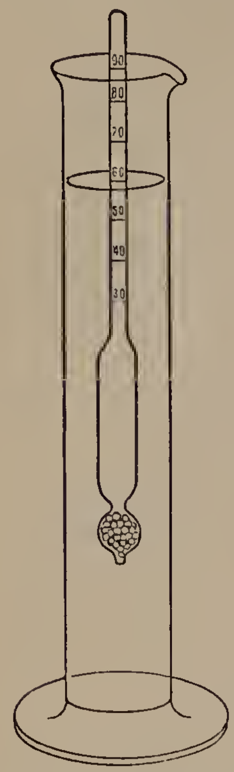

Fig. 77. Finding the density of gasolene.

NOTE. Commercial hydrometers are often graduated according to the **Baumé Scales.** The scales are divided into divisions called **degrees** (°); the scale for liquids lighter than water begins at 10°, which corresponds to a specific gravity of 1, and increases as the specific gravity decreases. *Degrees Baumé can be converted into specific gravity readings by the following equation.*

$$\text{Specific Gravity} = \frac{140}{130 + \text{Degrees Baumé}}$$

Commercial gasolene (petrol, motor spirit) ranges from 50° Bé to 90° Bé and over.

b. Volatility. This is probably the most important single test of gasolene. We may roughly compare the volatility of various samples of gasolene and of kerosene as follows: Place ten drops of gasolene on a watch glass and set the glass on a beaker half filled with boiling water.

CAUTION. Gasolene vapor is inflammable. Keep it away from all flames.

Note the length of time required for its complete evaporation. Repeat the experiment, using 10 drops of kerosene. *Compare the volatility of gasolene and kerosene.*

c. Combustion of gasolene. Warm a wide-mouth bottle and then pour into it a drop of gasolene. Cork the bottle and shake it vigorously. Remove the cork and bring a lighted splint to the mouth of the bottle. If there is no decided result, remove the splint, add another drop of gasolene and repeat the operation. Continue in this way to add gasolene, one drop at a time, until the vapor burns with explosive violence. *What was in the bottle besides gasolene? What is meant by an "explosive mixture"?*

d. Flashing point of kerosene. Arrange a porcelain dish on a sand bath as shown in figure 78. Fill the dish to within half a centimeter of the top with kerosene and adjust the thermometer in the center of the liquid so that it almost reaches the bottom of it but does not quite touch the dish. Put the Bunsen burner under the center of the dish and allow the apparatus to get warm. From time to time apply a little test flame by passing it slowly across the dish about a centimeter above the liquid and just in front of the thermometer.

Continue in this way until the vapor above the liquid ignites with a slight flash. The lowest temperature at which a liquid does this, as shown by a thermometer, is called the **flashing point.**

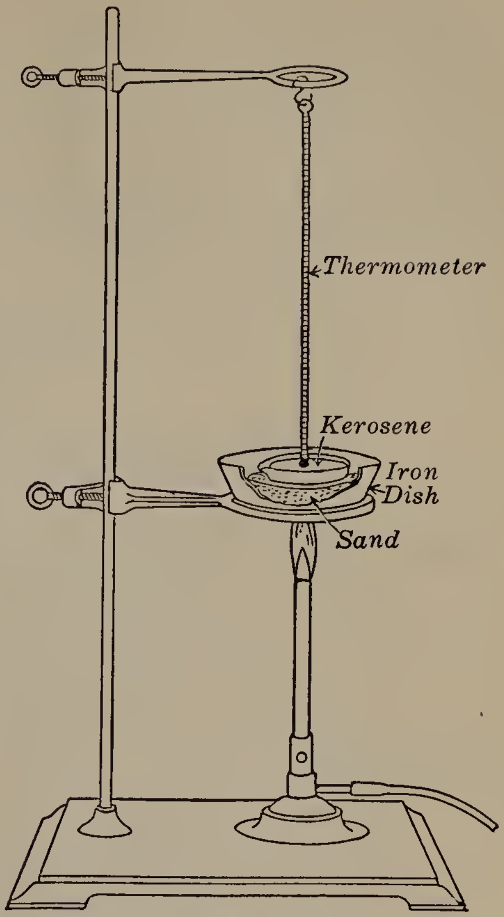

Fig. 78. Apparatus for determining the flash point of kerosene.

Repeat the experiment several times to make sure that you have found the lowest temperature at which the flash can be obtained.

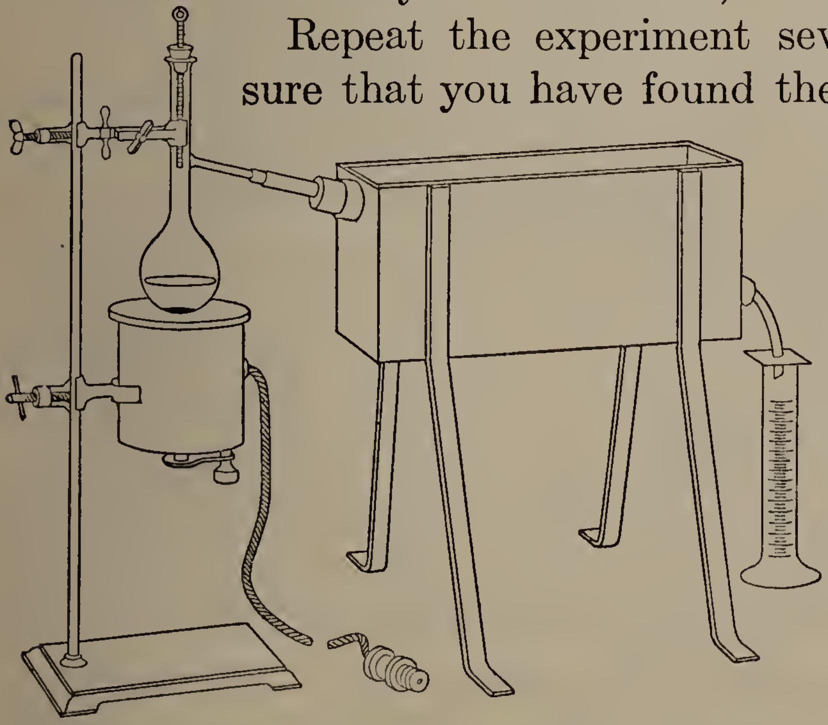

Fig. 79. Finding the boiling point and dry point of gasolene.

Note. Most of the states have laws which forbid the sale of oil for illuminating purposes which has a flashing point lower than about 110° F.

Optional work. In testing gasolene, it is sometimes necessary to determine the boiling point and the dry point. A very convenient form of apparatus, which makes use of an electric heater, is shown in figure 79. The technical details of this operation will be found in Technical Papers Nos. 166 and 214 of the Bureau of Mines — Methods of Testing Motor Gasolene, *E. W. Dean.*

EXPERIMENT 48

PREPARATION AND PROPERTIES OF ALCOHOL

Apparatus. — A 2-liter bottle or flask. Wide-mouth bottle with 2-hole stopper. Drying tube. Boiling flask with side tube. Tripod and asbestos gauze. Ring stand and clamp. Liebig condenser. Test tubes. Bunsen burner. Thermometer. Watch glass. Rubber tubing. Glass tubing.

Materials. — Corn sirup or molasses. Yeast cake. Limewater. Sticks of sodium hydroxide. Glass beads. Asbestos wool. Iodine. Sodium hydroxide solution. Litmus papers.

a. Fermentation.* Dissolve about 200 cc. of corn sirup or molasses in 2 liters of water in a large bottle or flask (Fig. 80). Crumble a yeast cake and mix it with water, making a thin paste. Then add it to the sugar solution. Connect the bottle with another containing limewater, as shown in the figure. The bulb tube contains sticks of sodium hydroxide to absorb any carbon dioxide from the air which might affect the limewater. Set the apparatus aside in a warm place (about 30° C.) for 2–3 days.

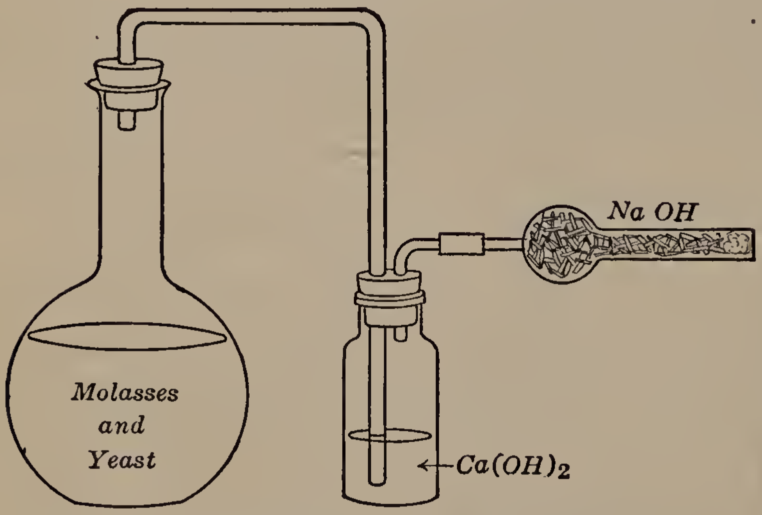

Fig. 80. Fermentation of molasses in the laboratory.

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* This part of the experiment may well be done by the instructor or a small group of students for the whole class.

b. Distillation. After the sugar solution has fermented, pour off the clear liquid so that each student or group of students gets about 150–200 cc. The apparatus for separating the alcohol from the solution may be set up as in figure 81 or with the condenser in an upright position as shown in figure 82. To prevent bumping, it is well to add a few glass beads or pieces of red-hot pumice stone to the boiling flask. In connecting the condenser to the water supply, be sure to have the cold water enter the jacket at the lower tube.

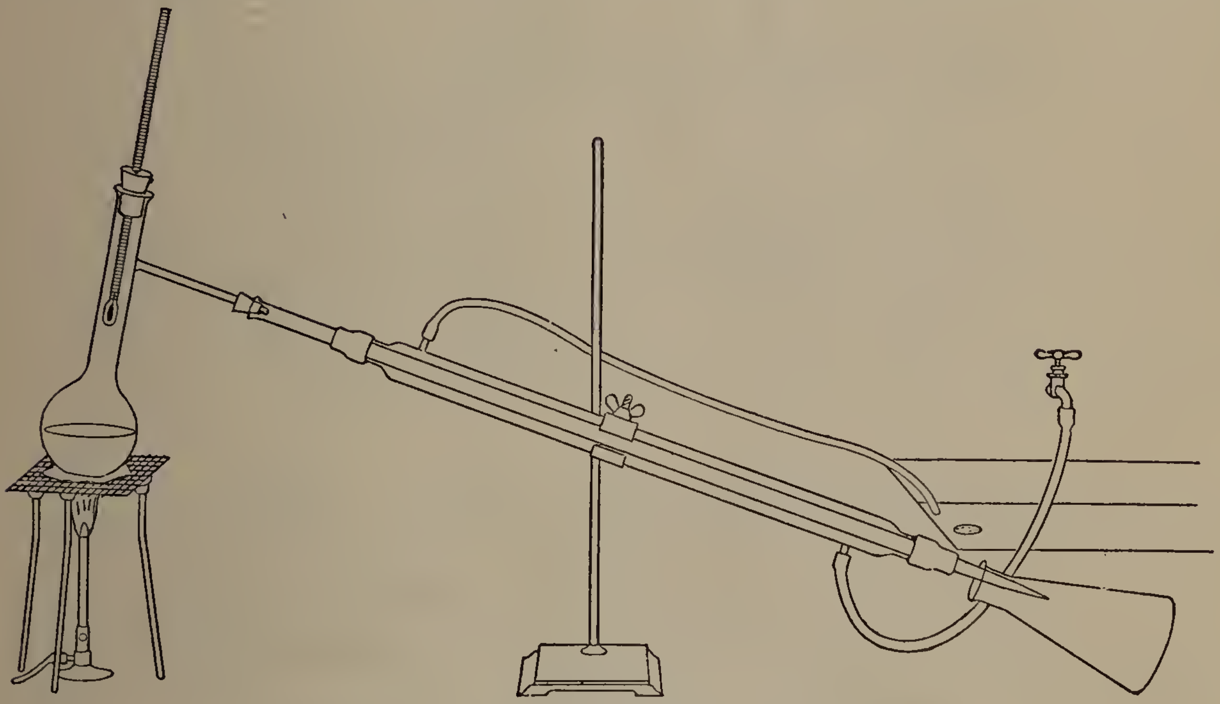

Fig. 81. Fractional distillation of alcohol.

Record the temperature at which the first few drops of liquid appear in the condenser and again when about one-half of the liquid has distilled over.

c. Properties. As soon as a few drops of the distillate have been collected, pour them on a watch glass. *Note the odor of the distillate.* Test it with litmus papers. Saturate a bit of asbestos wool with the liquid and try to ignite it. To recognize one product of burning alcohol, hold a cold wide-mouthed bottle over the flame. Close the bottle, pour in a little limewater, and shake the contents. *Make an equation for the combustion of alcohol* (C_2H_5OH).

d. Test. Add 3–4 cc. of iodine solution to about 5 cc. of the distillate and then add sodium hydroxide solution until the brown color disappears. Warm the mixture and set it aside for a few minutes. The precipitate is **iodoform** (CHI_3) and shows the presence of ethyl alcohol. *Record the color and odor.*

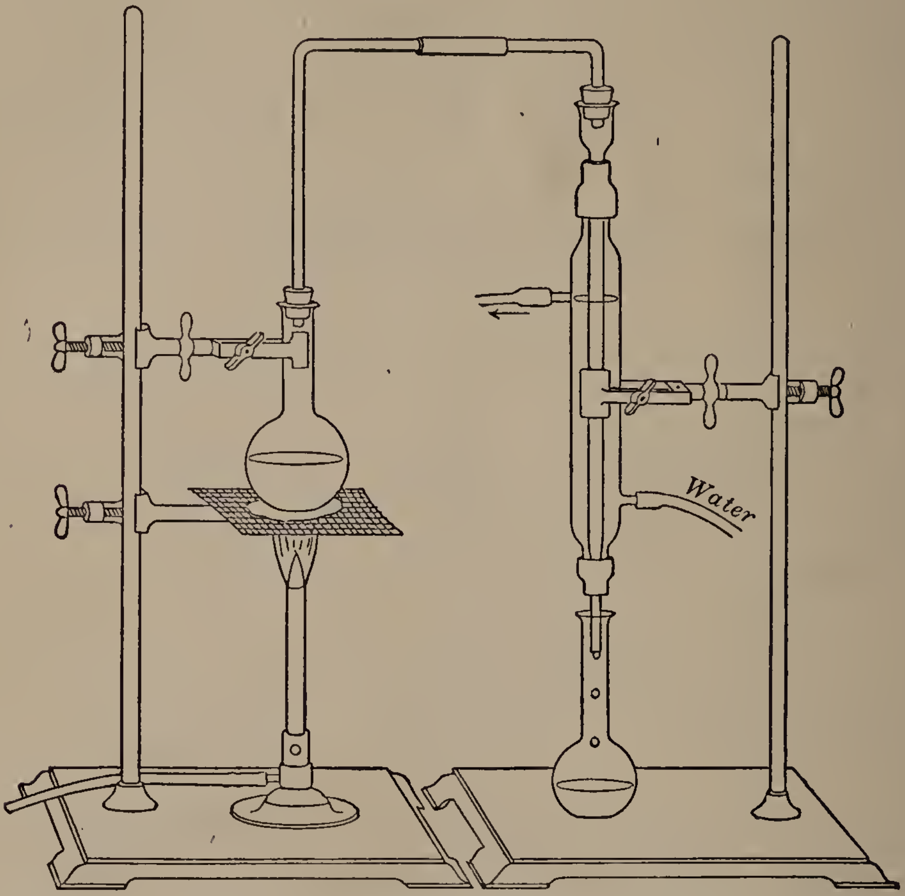

Fig. 82. Fractional distillation with an upright condenser.

Fig. 83. Testing for methyl alcohol with a hot copper coil.

e. ***Summarize*** *the properties of alcohol which you have observed. Name two substances which result from the fermentation of sugar.*

Optional work. Methyl alcohol (CH_3OH) is sometimes used as a substitute for ordinary alcohol. There is no simple direct test for methyl alcohol, but there is for formaldehyde (HCHO), into which it may easily be converted.

Mix about 1 cc. of methyl alcohol with about 6 cc. of water in a test tube which is kept cold by standing in a bottle of cold water. Heat a coil of copper wire (Fig. 83) to a dull red in the upper Bunsen flame and then plunge the hot coil quickly into the mixture in the tube. Withdraw the coil and dip it into water. Repeat the operation six times. In this way most of the alcohol will be changed into formaldehyde. Compare the odor of this mixture with that of the formaldehyde in the stock-room bottle.

EXPERIMENT 49

THE MAKING OF SOAP — ITS USES

Apparatus. — Evaporating dish. Tripod and asbestos gauze. Bunsen burner. Stirring rod. Test tubes and rack.

Materials. — Alcohol. Vegetable oil (olive, cotton seed, or cocoanut). Sodium hydroxide solution (20%). Calcium chloride solution. Kerosene.

a. Preliminary. Sodium hydroxide (NaOH) is one of the strong bases (an alkali). When fats or vegetable oils are boiled with it, they are saponified, *i.e.* converted into soap. These fats are chiefly glyceryl esters of organic acids. On boiling, the fat is decomposed by the sodium hydroxide into glycerin ($C_3H_5(OH)_3$) and the sodium salt of the organic acid, which is soap. The following equation represents such a reaction:

$$\underset{\text{Glyceryl stearate}}{\overset{\text{FAT}}{(C_{17}H_{35}COO)_3C_3H_5}} + \overset{\text{ALKALI}}{3\ NaOH} \rightarrow \underset{\text{Sodium stearate}}{\overset{\text{SOAP}}{3\ C_{17}H_{35}COONa}} + \overset{\text{GLYCERIN}}{C_3H_5(OH)_3}$$

b. Preparation. Add 10 cc. of alcohol to about 2 teaspoonfuls of oil (olive, cottonseed, or cocoanut) in an evaporating dish. Heat the dish with a small flame which barely touches the dish. Then add 5 cc. of a 20 per cent solution of sodium hydroxide and continue heating and stirring the contents constantly until the odor of alcohol is no longer perceptible (Fig. 84). The pasty mass which remains in the dish is the soap mixed with glycerin.

NOTE. To save time in this experiment alcohol is used as a common solvent for the oil and the alkali. In commercial work it is not used.

c. Uses. Wash your hands with a little of the soap you have just made. *Does it lather readily?* When too much alkali has been used, the soap makes the hands rough. An excess of oil makes the soap greasy. *What about the quality of your soap?*

Make a half test-tubeful of soap solution, add to it half as much calcium chloride ($CaCl_2$) solution, and shake the liquid. An insoluble calcium soap is formed. This insoluble soap, which is formed whenever soap is used with "hard" water, is useless for cleaning purposes.

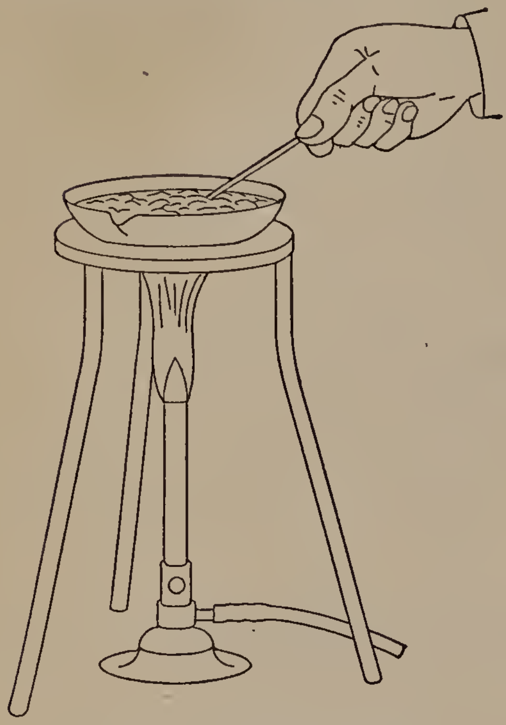
Fig. 84. Making soap.

Put 1 cc. of kerosene into 10 cc. of water in a test tube and shake the mixture. Note that the oil is broken up into tiny globules which form an **emulsion.** Let the test tube stand for a minute to see whether the emulsion remains; then add about 2–3 cc. of soap solution to the mixture and shake again. *What effect does the soap have on the permanency of the emulsion?* In this same way soap forms an emulsion with the oil which makes dirt cling to the fibers of clothing. This action loosens the dirt so that it can be easily removed by mechanical washers.

Optional work. Soap can be made from waste fat. Even though the fat is dirty and rancid, it can be used by melting and straining it through cheesecloth. Weigh the fat and also weigh out about one-third as much commercial lye (Babbitt's). Dissolve the lye in water. Heat the fat in an iron kettle and slowly pour in the lye solution with constant stirring. Some care is required to see that the liquid does not boil over. After it has boiled for 30 minutes, add water to make up for that which has boiled away and then stir in common salt, about twice the weight of the lye used. When cold, remove the layer of soap on top. The soap may be warmed and allowed to cool in small pasteboard boxes so as to form cakes.

EXPERIMENT 50

Removal of Spots and Stains

Apparatus. — Beakers. Test tubes and rack. Bunsen burner.
Materials. — Samples of various kinds of cloth (cotton, wool, silk) spotted with grease, sugar, ink, iron rust, etc. Carbon tetrachloride, turpentine, oxalic acid, Javelle water, etc.

a. Preliminary. The simplicity of the cleansing process varies with the character of the stain. For example, the removal of a grease spot from cloth is merely a process of solution and absorption; but the removal of a fruit stain or of an ink spot from cloth often involves a careful study of the cloth itself as well as of the various bleaching reagents.

In general there are three methods of treatment: (1) **Warm water** will remove sugar, glue, blood, if not too old; (2) **Gasolene, carbon tetrachloride**, or **turpentine** will act as a solvent for grease and paint and so can be used to remove such spots; (3) **Bleaching agents**, such as oxalic acid or citric acid, followed by bleaching powder may be used to remove ink spots. Of course it is quite important to remember that gasolene vapor is very inflammable and should never be used in the vicinity of a flame. Some absorbing material must be placed beneath the goods to be cleaned in order that the grease may be washed through by the gasolene. Otherwise, rubbing a little gasolene on a spot merely spreads the grease over a larger area. In using bleaching agents it is necessary to remove with dilute ammonia water the traces of such agents, which might rot the fibers of the cloth.

It is always well to treat the stain on a small sample of the goods before trying to remove the spot or stain from a garment. In general greater care must be exercised with woolen and silk goods than with cotton, and especially with colored goods.

The following table will be useful for reference:

Stain	Removed by
Sugar, glue, blood	Warm water
Grease	Carbon tetrachloride or gasolene; followed by warm soapsuds and ammonia water
Paint, varnish	Turpentine (except on silk)
Tar, pitch	Soap and oil; followed by turpentine
Inks	Warm oxalic or tartaric acid (20%) or salt and lemon
Iron rust	Warm oxalic or citric acid (10%)
Fruit, tea, and coffee	Hot water. Javelle water *
Acids	Ammonia water to neutralize the acid; then wash with water
Grass	Sponge with alcohol or ammonia water

b. Practice. Take samples of 3 or 4 different kinds of cloth and stain them with ink, acid, sirup, and oil. Treat each sample according to the methods just described. Cut each sample down to some convenient but uniform size and mount it with a label in your notebook. Show a sample of the piece of cloth before and after treatment. Also describe briefly just what the treatment was in each case.

Optional work. Find out the composition of the two-solution ink eradicator, of the French dry-cleaning fluid, of a reagent for mildew stain, and of a paint and varnish remover. (Consult *Smith & Mess'* Laboratory Study of Chemistry.)

* Javelle water is made by dissolving a pound of washing soda ($Na_2CO_3 \cdot 10\ H_2O$) in a quart of boiling water. Add a quarter of a pound of chloride of lime ($CaOCl_2$) to a quart of hot water. Mix the two solutions and decant the clear solution of sodium hypochlorite ($NaOCl$) for use.

EXPERIMENT 51

TEXTILE FIBERS

Apparatus. — Compound microscope. Test tubes and rack. Bunsen burner.

Materials. — Samples of cotton, wool, silk, and linen cloth. Samples of cotton and wool mixed goods, cotton and linen mixed goods. Sodium hydroxide solution (20%). Alcoholic solution of fuchsin. Dilute ammonium hydroxide. Nickel hydroxide solution. Dilute hydrochloric acid.

a. Microscopic study of fibers. Study carefully the appearance of fibers of cotton, wool, silk, and linen under the microscope. (Compare with figure 174 on page 285 of B. & C.)

b. Cotton and wool. Place in separate test tubes a sample (3 cm. × 3 cm.) of cotton cloth, woolen cloth, and mixed goods. Cover each with sodium hydroxide solution (20%) and boil for 2–3 minutes. Add water when necessary to keep the cloth covered with liquid.

CAUTION. Take great care not to let the hot alkali spatter over on to the flesh.

Which one of these fibers is completely soluble in sodium hydroxide solution?

c. Cotton and linen. Wet a sample of cloth (part linen) with a 1 per cent alcoholic solution of fuchsin, wash well in water, and then moisten with ammonium hydroxide. The linen (flax) fibers in the cloth will be colored red, while the cotton remains uncolored.

d. Wool and silk. Put strips of wool and silk in separate test tubes and cover each with a solution of nickel hydroxide.*

---

* **Nickel hydroxide** solution may be prepared by dissolving 5 g. of nickel sulfate in 100 cc. of water and then adding a solution of sodium hydroxide until the nickel hydroxide is completely precipitated. Wash the precipitate well and dissolve it in 25 cc. of concentrated ammonium hydroxide. Then add 25 cc. of distilled water.

After 2 minutes remove the residue and boil for another 2 minutes in a 1% solution of hydrochloric acid. *Which of the samples remains undissolved?*

e. Silk, wool, and cotton. *How would you prove the presence of silk, wool, and cotton in a sample?*

Optional work. Weigh to a centigram a sample (10 cm. × 10 cm.) of some mixed goods. First remove the sizing by boiling the cloth for 10 minutes in a 1 per cent solution of hydrochloric acid. Wash and dry the sample and reweigh to determine the per cent of sizing. Then proceed to separate the various fibers as just described and calculate the per cent of each kind of fiber. Mount neatly in your notebook a sample (3 cm. × 3 cm.) of the cloth analyzed.

How would you distinguish between real silk and artificial silk?

EXPERIMENT 52

Components of Foods

Apparatus. — Test tubes and rack. Bunsen burner. Mortar and pestle. Large beaker. Funnel. Crystallizing dish. Iron spoon.

Materials. — Starch. Iodine solution. Glucose. Fehling's solution (two solutions). Concentrated nitric acid. Concentrated ammonium hydroxide. Egg. Cornmeal. Peanuts. Oatmeal. Ether. Filter papers.

a. Starch. Put a pinch of starch into a test tube and fill the tube half full of water. Shake the contents and boil. *Has the starch changed?* Cool the liquid in running water, and, as soon as it is cold, divide it into two parts. To one add a drop of iodine solution (iodine in potassium iodide). The blue color which is produced is the iodine **test for starch.**

b. Glucose. Dissolve about 1 cc. of glucose in 10 cc. of water. Add 5 cc. of an alkaline solution of copper sulfate (Fehling's solution) and boil for a few minutes. The red precipitate (cuprous oxide, Cu_2O) is the characteristic **test for glucose.**

c. Protein. Place a few bits of the white of a hard-boiled egg in a test tube and add a few drops of concentrated nitric acid. Wash off the acid with water and pour off the liquid. Then pour a few drops of ammonium hydroxide on the egg. The yellow color is a **test for proteins** (nitrogenous compounds).

d. Fats and oils. Half fill a test tube with cornmeal or crushed peanuts and pour in enough ether or benzine (away from flames) to cover the solid to a depth of a centimeter. Stand the test tube in a beaker of hot water and shake the contents of the test tube occasionally. Filter and let the filtrate evaporate spontaneously in a crystallizing dish. The fat or oil extracted remains in the dish.

e. Mineral matter. Place about half a teaspoonful of oatmeal in an old iron spoon and heat it (under the hood) until no more fumes are given off, then heat it strongly to burn off the carbon. The white ash contains various mineral compounds.

f. Examination of a food. Apply these tests to one or more foods, such as lean meat, bread, beans (dried), and cheese. Record your results in tabular form.

Optional work. Water is one of the important components in foods. Devise some method of determining the per cent of water in some article of food, such as an apple or a potato. When your method is approved by the instructor, carry it out and compare your results with those given on page 289 in B. & C.

EXPERIMENT 53

Food Components of Milk

Apparatus. — Evaporating dish. Short stirring rod. Steam bath. Tripod. Bunsen burner. Ring stand. Horn pan balance and weights. Test tubes and rack. Funnel.

Materials. — Sample of milk (from home). Acetic acid. Junket tablets. Fehling's solution. Concentrated nitric acid. Concentrated ammonium hydroxide.

a. Per cent of solids and water. To determine the percentage of water in milk, we have only to evaporate to dryness a known weight of milk. During this process a scum forms over the surface of the liquid, which retards evaporation, and so it is well to use a short stirring rod occasionally to break up the scum.

Weigh a small, clean evaporating dish together with a short stirring rod and then pour into it about 20 cc. of milk and weigh again. Evaporate over a steam bath (Fig. 85) to dryness. As soon as the residue is perfectly dry, wipe dry the outside of the dish, cool, and reweigh. From these results compute the percentages of solids and water in the sample of milk taken. (Cow's milk usually contains not less than 12 per cent of solids.)

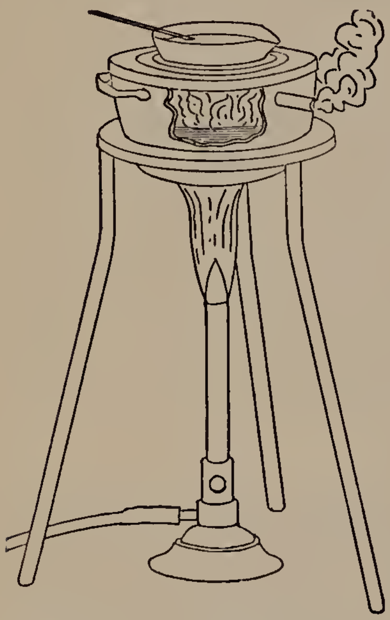

Fig. 85. Evaporating milk over water bath.

b. Protein. The principal protein in milk is casein, which is used in the manufacture of cheese. Add 2–3 drops of acetic acid (vinegar may be used) to 10 cc. of milk. When thoroughly mixed and allowed to stand, the casein separates in the form of a curd. When milk sours, the lactic acid formed in the milk causes a natural curdling. Prove that the curd is a protein (Exp. 52).

Another method of separating the casein is by means of rennet. "Junket" tablets contain dried rennet. Dissolve about one-half of such a tablet in water and add the solution to about 100 cc. of warm milk. Again the casein will separate as a curd.

There is another protein in milk which remains in solution when the casein is separated either by an acid or by rennet. This is **albumin**, which is much like the white of egg. Filter off the milk curd (casein) and boil the clear liquid (whey). This coagulates the albumin in the form of white flakes. Prove that the tough scum which forms on the surface of boiled milk is coagulated albumin (a protein).

c. Sugar of milk (lactose). The filtrate left after the casein and albumin have been coagulated and removed contains the sugar of milk. By evaporation of this filtrate we obtain a residue. The taste shows that this sugar is only about one-sixth as sweet as cane sugar. Test it with Fehling's solution.

Optional work. The most valuable component of milk is doubtless the **butter fat.** The most practical method of determining the per cent of butter fat is the **Babcock test.** In this process a definite quantity (17.6 cc.) of milk is treated with sulfuric acid (17.5 cc., Sp. Gr. 1.83) which dissolves the casein of the milk and thus facilitates the separation of the fat. This mixture is then whirled in a centrifugal machine, and the fat collects on the surface. The necessary apparatus is shown in figure 86. The test bottle is so made that the graduations on the neck indicate the percentage of fat directly. Further directions are furnished with the apparatus.

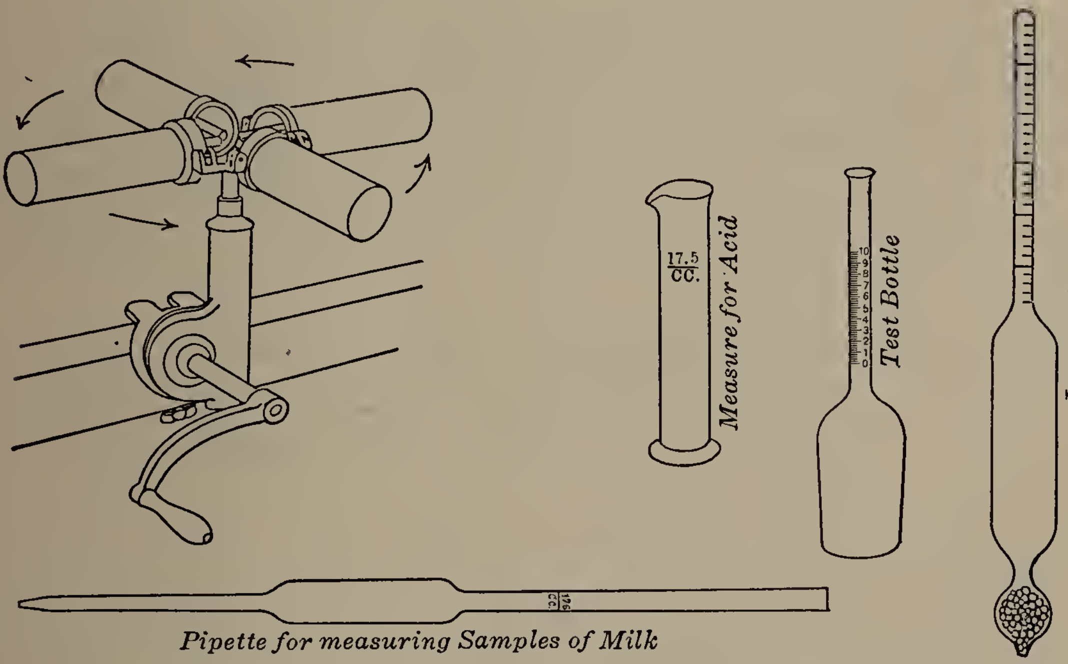

Fig. 86. Milk-testing outfit. Centrifuge. Babcock test bottles and lactometer.

By using the Babcock test and by determining the specific gravity with a lactometer it is possible to estimate approximately the other solids in milk in addition to the fat. The Babcock formula, which is based on experimental results, is as follows:

$$\text{Solids-not-fat} = \frac{\text{Lactometer}}{4} + 0.2\ \text{Fats}$$

The lactometer commonly used (Quevenne) is so graduated that the scale represents only the second and third decimal places of the specific gravity. For example, if it reads 34 the specific gravity is 1.034; and if the percentage of fat is 3.5 per cent, then the per cent of **solids-not-fats** is $\frac{34}{4} + 0.2 \times 3.5$ or **9.2**.

EXPERIMENT 54

Testing Foods for Adulterations

Apparatus. — Iron spoon. Bunsen burner. Wooden splint. Small flask. Beaker. Tripod. Asbestos gauze. Test tubes.

Materials. — Creamery butter and oleomargarine. 1 pint of sweet milk (from home). Concentrated sulfuric acid with ferric chloride solution (25 cc. acid and 1 cc. $FeCl_3$ sol.). Formaldehyde. Cracked ice.

a. Butter and its substitutes. Many of the substitutes for butter are wholesome and much cheaper than real butter. But these substitutes should not be sold as butter. The following simple tests will serve to distinguish butter from its substitutes.

Foam test. Melt a lump of the so-called butter in a large iron spoon over a small flame. Genuine creamery butter will boil quietly with the production of considerable froth or foam. This is caused by the escape of steam from the butter. Oleomargarine and renovated butter bump and sputter and make more noise, producing little foam. Test a piece of real butter and compare its action with that of a substitute.

Waterhouse test. Place about 50 cc. of sweet milk in a small flask and set in hot water to heat. When it is almost boiling, add 4–5 g. (about a teaspoonful) of the sample and stir it with a wooden stick until it melts. Then place the flask in ice water and continue stirring until the fat solidifies. If the sample is **oleomargarine**, the fat can be collected into one lump which can be removed with the stick. **Butter fat** cannot be so collected, but is more or less emulsified with the milk. If the sample is **renovated butter**, it will tend to collect, when the stirring is stopped, as a film on the top of the milk.

b. Formaldehyde in milk. The use of this preservative in milk is especially pernicious because milk is so largely used as food for babies and children. Formaldehyde is one of the most poisonous preservatives found in foods. Milk should be regarded with suspicion if it takes too long to sour.

Bring a pint of milk from home or from a store and have the instructor add 2–3 drops of formaldehyde. Label the bottle and set the milk aside. Record how long it takes the milk to sour.

To test suspected milk for formaldehyde fill a test tube one-third full of the milk. Holding the tube in an inclined position, carefully pour down the side of the tube a little concentrated sulfuric acid to which has been added a drop of ferric chloride solution. The milk will rest upon the surface of the acid and if formaldehyde is present, a violet color forms where the acid and milk come into contact. Apply this test to the milk to which formaldehyde was added.

Optional work. Although the subject of food inspection and analysis is one of vital interest to the community, yet it is one which generally requires considerable technical training and experience. Nevertheless there are some simple tests which can be easily applied. The student is referred to the handy little book, by *Lewis B. Allyn*, Elementary Applied Chemistry, and to the larger book on the subject, by *A. E. Leach*, Food Inspection and Analysis.

EXPERIMENT 55

Sodium Carbonate and Bicarbonate

Apparatus. — Test tubes. 1-hole stopper to fit test tube. Ring stand and clamp. Bunsen burner. Beakers. Tripod and asbestos gauze. Glass tubing. Horn pan balance and weights.

Materials. — Sodium bicarbonate (baking soda). Limewater. Potassium bitartrate (cream of tartar).

a. Effect of heating the bicarbonate. Put about 2–3 g. of sodium bicarbonate ($NaHCO_3$) into a test tube and clamp it so that the mouth is inclined slightly downwards. Arrange a delivery tube so that it leads into a test tube containing limewater ($Ca(OH)_2$) (Fig. 87). Warm the bicarbonate gently with a small flame, taking care not to heat the test tube hot enough to color the flame yellow.

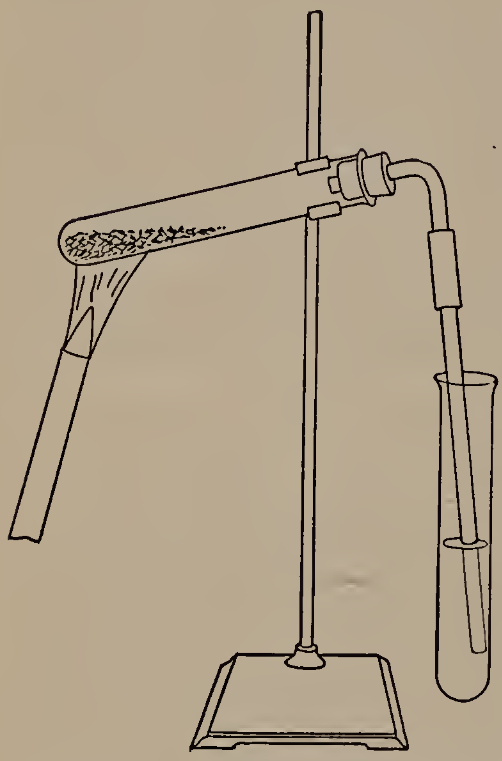

Fig. 87. Heating sodium bicarbonate and passing the gas into limewater.

What gas is evolved? What collects on the cool part of the test tube? To part of the residue left in the tube, add an acid. *What is the result? What is the residue? Write the equation for the action of heat on sodium bicarbonate. Compare the taste of the bicarbonate with that of the residue.*

b. Baking powder. Weigh out 2 g. of potassium bitartrate (cream of tartar, $KHC_4H_4O_6$). Calculate the amount of sodium bicarbonate required from the following equation:

$$\underset{\text{cream of tartar}}{KHC_4H_4O_6} + \underset{\text{baking soda}}{NaHCO_3} \longrightarrow \underset{\text{Rochelle salt}}{KNaC_4H_4O_6} + H_2O + CO_2\uparrow$$

Weigh out this amount of sodium bicarbonate and mix the two salts thoroughly on paper.

Put half of this mixture into a beaker half full of *cold* water and the other half into another beaker half full of *hot* water. *What is the gas which escapes? What is the difference between the action of cold and hot water on this mixture? Why is water needed in this chemical reaction?*

Optional work. Sodium bicarbonate may be prepared according to the **Solvay process.** Dissolve 10 g. of powdered ammonium carbonate in 100 cc. of cold concentrated ammonium hydroxide. Shake the mixture to secure a solution and then saturate the solution with sodium chloride by shaking the liquid vigorously with finely pulverized salt.

Pour off the clear solution into a bottle and pass slowly a stream of washed carbon dioxide gas through it until a copious precipitate of sodium bicarbonate is produced. Filter, and dry the precipitate by pressing between filter papers.

EXPERIMENT 56

ANALYSIS OF BAKING POWDER

Apparatus. — Test tubes and rack. Bunsen burner. Evaporating dish. Funnel. Beaker (small). Stirring rod. Ring stand and asbestos gauze.

Materials. —Sample of baking powder (2 tablespoonfuls, from home). Iodine solution. Barium chloride solution. Dilute hydrochloric acid. Concentrated sulfuric acid. Concentrated nitric acid. Ammonium molybdate solution. Sodium hydroxide solution. Filter papers.

a. Preliminary. Sodium bicarbonate is an essential ingredient of all baking powders. *How could you prove its presence?* The other necessary ingredient is some solid substance which is weakly acidic, such as cream of tartar, calcium acid phosphate, or alum. A small amount of starch or flour is usually added to keep the mixture dry. The different kinds of baking powder vary according to the kind of acid agent present. Some powders have a mixture of two or even of all three of these acid-forming ingredients.

Place about 10 g. of a baking powder in a beaker and add about 50 cc. of water. Stir the mixture well until all the gas is evolved. Then filter and test the filtrate and residue for the various ingredients as follows:

b. Starch. *Will the starch be found in the filtrate or in the residue?* Test for starch with one drop of iodine solution as in Exp. 52.

c. Sulfates. Ordinary **alum** is a double salt of aluminum sulfate and potassium sulfate ($K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24\,H_2O$). **Ammonium alum** is also a double salt of ammonium sulfate and aluminum sulfate ($(NH_4)_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24\,H_2O$). Thus it will be seen that if a baking powder contains alum, the filtrate will contain sulfates. Test the filtrate with barium chloride solution and dilute hydrochloric acid as in Exp. 36.

d. Tartrates. Pour about 5 cc. of the filtrate from (a) into an evaporating dish and add 5 drops of concentrated sulfuric acid. Evaporate to dryness over a small flame. Charring and the odor of burnt sugar indicate the presence of a tartrate.

e. Phosphates. If the baking powder contains calcium acid phosphate, it will be found in the filtrate from (a). To test for a phosphate, take a few cubic centimeters of the filtrate and acidify with a few drops of nitric acid. Warm the mixture and add a few drops to 5 cc. of ammonium molybdate solution ($(NH_4)_2MoO_4$). Set it aside. A bright yellow precipitate indicates a phosphate.

f. Ammonium salts. If the baking powder contains ammonium alum, we can detect it by pouring about 5 cc. of the filtrate into a test tube and adding the same volume of sodium hydroxide solution. If heated to boiling, ammonia gas is evolved.

g. Calcium and aluminum. Since a baking powder containing sulfates always contains aluminum and one containing phosphates always contains calcium, we need not make special tests for these metals here.

In making the report of your chemical analysis, state *where* *you obtained it, the name of the manufacturer, exact name of brand, what you tested for and how, and what you found present.*

Optional work. Compute the cost of making tartrate baking powder according to the following formula: 1 lb. sodium bicarbonate, 2 lb. cream of tartar, 1 lb. cornstarch. Use the prices given in a trade catalogue. Would it be worth while to prepare baking powder for home use? Why should it be kept in air-tight containers?

Look up the formulas of some other baking powders and compute the cost of ingredients.

EXPERIMENT 57

Hydrolysis of Salts

Apparatus. — Test tubes and rack. Stirring rod.

Materials. — Litmus papers (red and blue). Copper sulfate, aluminum sulfate, ferric chloride, sodium chloride, potassium nitrate, sodium carbonate, and potassium carbonate.

a. Preliminary. Not all salt solutions react neutral when tested with litmus paper. This is due to the fact that water is itself very slightly dissociated into H^+ and OH^-. The action of the ions of water on a dissolved salt is called hydrolysis. When the ions of the salt unite with the ions of water to form the undissociated molecules of a weak base, an equivalent amount of hydrogen ions (H^+) is set free and the solution gives an **acid** reaction with litmus. When the ions of the salt unite with the ions of water to form the undissociated molecules of a weak acid, an equivalent amount of hydroxyl ions (OH^-) is set free and the solution gives a **basic** reaction with litmus.

Note. Remember that the reddening of blue litmus shows H^+ and the turning blue of red litmus shows OH^-.

b. Testing salt solutions with litmus. Dissolve about 0.5 g. of each of the following substances in 10 cc. of distilled water in separate test tubes: copper sulfate ($CuSO_4$), sodium chloride

(NaCl), sodium carbonate (Na_2CO_3), aluminum sulfate ($Al_2(SO_4)_3$), ferric chloride ($FeCl_3$), potassium nitrate (KNO_3), and potassium carbonate (K_2CO_3).

Test each solution with litmus paper by putting a small drop of the liquid on the paper with a clean glass rod. *Divide these solutions into three classes:* (1) those which give an *acid* reaction, (2) those that give a *basic* reaction, and (3) those that are *neutral.* Take one case from each of these three classes and *explain in terms of the ionic theory the reaction of the solution toward litmus. Write equations to illustrate hydrolysis in classes* 1 *and* 2.

EXPERIMENT 58

FLAME TESTS

Apparatus. — Bunsen burner. Platinum wire (iron or nichrome) mounted in a glass tube. Test tubes. Cobalt glass (2 or 3 squares).

Materials. — Concentrated hydrochloric acid. Chlorides of sodium, potassium, lithium, calcium, strontium, and barium. Unknowns.

a. Preliminary. There are certain metals which are easily detected, in whatever salt they may occur, because of a characteristic color which each imparts to the Bunsen flame. This furnishes a convenient and often a very delicate qualitative test for these metals. We shall study compounds of **sodium, potassium, lithium, calcium, strontium,** and **barium.** We may use a piece of platinum wire which has a little hook bent at one end and is melted into a short piece of glass tube at the other end. (Nichrome wire may be substituted here, and one end may be twisted around a match stick to serve as a handle.)

Hold the end of the platinum wire in the blue Bunsen flame. If the flame is colored by it, dip the wire in a little concentrated hydrochloric acid in a test tube and again hold it in the flame. Repeat this several times until the wise ceases to impart any color to the flame.

b. Sodium. Heat the end of the cleaned wire and quickly touch a tiny particle of sodium chloride. Hold the wire with the adhering particle in the lower part of the outer blue layer of the flame (Fig. 88) and *note the color of flame produced.* Examine the sodium flame through two thicknesses of blue (cobalt) glass. *What tint of light is absorbed by the blue glass and hence does not reach the eye?* Hold the wire in the flame until the color has nearly disappeared, then clean it as before by dipping in hydrochloric acid.

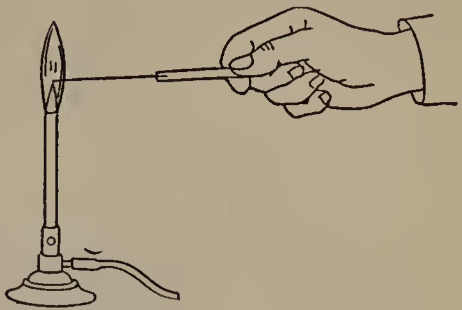
Fig. 88. Flame test for sodium, etc.

c. Potassium. When the wire is quite clean, heat it and touch a bit of potassium chloride. *Record the color imparted to the flame.* Hold two thicknesses of blue glass between the flame and the eye and observe whether the color is absorbed.

Mix together intimately a little sodium chloride and potassium chloride and plunge the hot wire into the mixture. *Record the color imparted to the flame. Why do you not see both of the characteristic colors?* Now examine the flame of the mixture through blue glass. *Which metal can you recognize in this way?*

d. Lithium, calcium, strontium, and barium. When the wire is thoroughly clean and imparts no color to the flame, plunge it into a little lithium chloride and hold it in the flame. *Note the characteristic color.*

Repeat this experiment with the chlorides of the next three metals (Ca, Sr, and Ba), and tabulate your results:

SUBSTANCE	FORMULA	COLOR OF FLAME	REMARKS

e. Test for unknown. Obtain from the instructor a numbered envelope containing an "unknown." This may contain one or two salts of these six metals. *Record the number of your unknown and identify it.*

Optional work. If a spectroscope (Fig. 89) is available, study the spectra of these metals and compare your results with those shown on the Spectrum Chart (facing page 310, B. & C.).

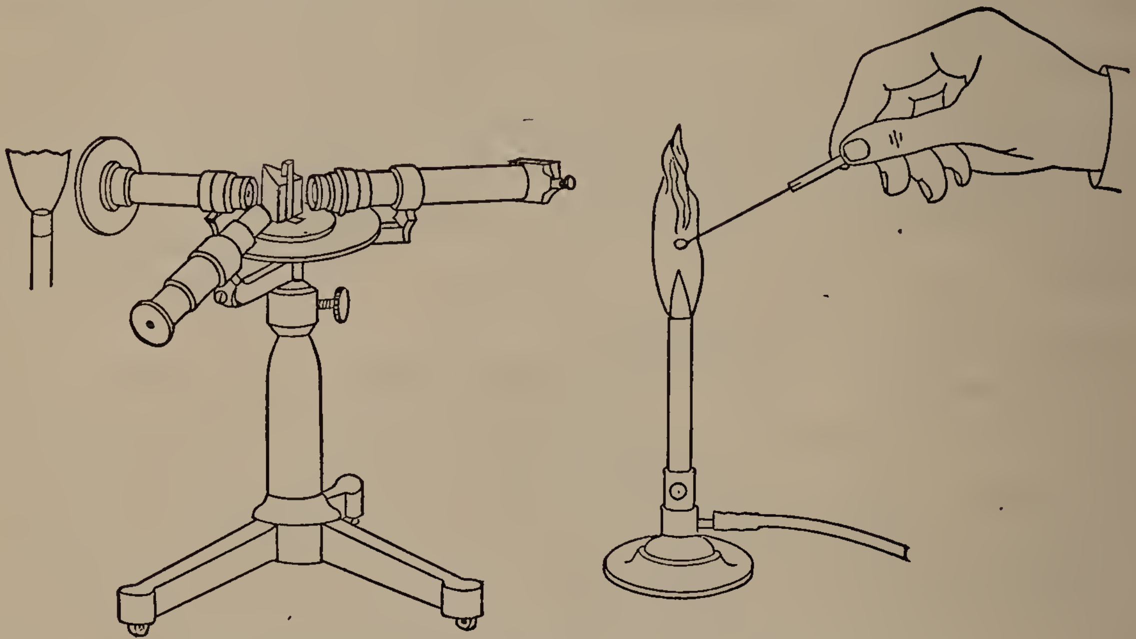

Fig. 89. Spectroscope used in spectrum analysis.

EXPERIMENT 59

CHLORINE — PREPARATION AND PROPERTIES

NOTE. If the laboratory is not provided with suitable hoods, this experiment should be done by the instructor.

Apparatus. — Flask with 2-hole stopper. Ring stand with large ring and clamp. Thistle tube. Bunsen burner. Pan or water bath. 4 wide-mouth bottles and glass plates. Pneumatic trough. Mortar and pestle. One hydrogen generator for class. Glass tubing.

Materials. — Manganese dioxide. Concentrated hydrochloric acid. Powdered antimony. Taper.

CAUTION. Chlorine is a poisonous gas. Avoid inhaling much chlorine. Breathing ammonia gives relief. This experiment should be done under a hood or in a place where there is a good draft.

a. Preparation. Set up the apparatus as shown in figure 90. Place about 10 g. of granular manganese dioxide in the generating flask and pour in about 25 cc. of concentrated hydrochloric acid. Rotate the flask so as to mix the contents thoroughly.

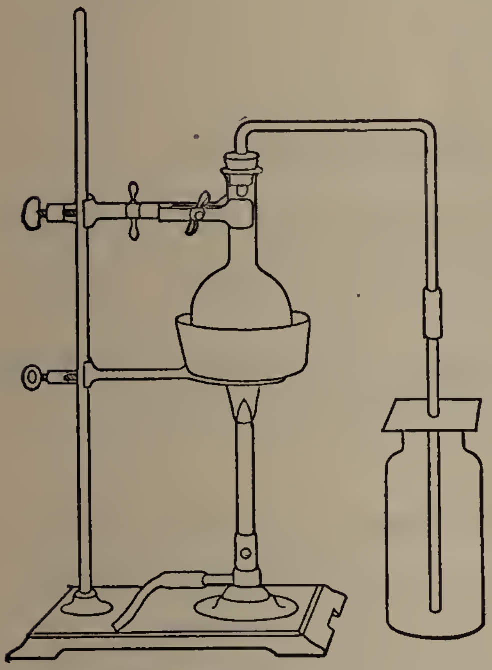

Fig. 90. **Preparation of chlorine.**

Heat the water under the flask to boiling. Fill 4 wide-mouthed bottles by the displacement of the air, holding a piece of white paper behind the bottles to see when they are full. *Note the color of the gas.* Cover the bottles with glass plates and set them aside.

b. Properties. ***Solubility in water.*** Slide one of the glass plates aside and fill one bottle of chlorine about one-third full of cold water. Cover it with the palm of the hand and shake the contents vigorously. Lower the bottle into a trough of water and remove the hand. *Compare the solubility of chlorine with that of hydrogen chloride* (Exp. 17).

Very Sol!

Action with metals. Grind up in a mortar a fragment of antimony and sprinkle some of the powdered antimony into a bottle of chlorine. *Name the product and write the equation. Is oxygen always necessary for combustion?*

Burns

Action with hydrogen. Introduce a jet of burning hydrogen into a bottle of chlorine. Blow gently across the mouth of the bottle and note the change of color. *Name the product and write the equation for the burning.*

Burns

Action with hydrocarbons. Lower a lighted taper or candle into a bottle of the gas by means of a wire handle. Blow gently across the mouth of the bottle. *Name two products of this action.*

Not
burns

c. ***Summarize*** *the properties of chlorine which you have observed in this experiment.*

Optional work. Since chlorine is prepared by the oxidation of the hydrogen in hydrochloric acid, we can use various oxidizing agents. Place a few crystals of potassium permanganate in a beaker and add 2–3 cc. of concentrated hydrochloric acid. Cover the beaker with a glass plate. After a few minutes examine the beaker and identify the gas.

Repeat the experiment using in turn powdered potassium chlorate, potassium dichromate, and lead dioxide, as the oxidizing agent.

EXPERIMENT 60

BROMINE — PREPARATION AND PROPERTIES

Apparatus. — Ring stand and clamp. Test tubes and rack. 1-hole stopper to fit test tube. Wide-mouth bottle. Bunsen burner. Glass tubing.

Materials. — Potassium (or sodium) bromide. Manganese dioxide. Concentrated sulfuric acid. Carbon disulfide. Chlorine water (saturate water with the gas).

a. Preparation. Fit a cork stopper to a test tube and bore a hole through it. Insert a delivery tube and set up the apparatus as shown in figure 91. Mix on a piece of paper about 1 cc. of potassium bromide (or sodium bromide) crystals with twice this bulk of manganese dioxide and put the mixture into the test tube. Add about 2 cc. of concentrated sulfuric acid to 1 cc. of water in a test tube and cool it. Then pour the acid on the mixture of potassium bromide and manganese dioxide. Insert the stopper carrying a delivery tube which extends to the bottom of an empty test tube standing in cold water.

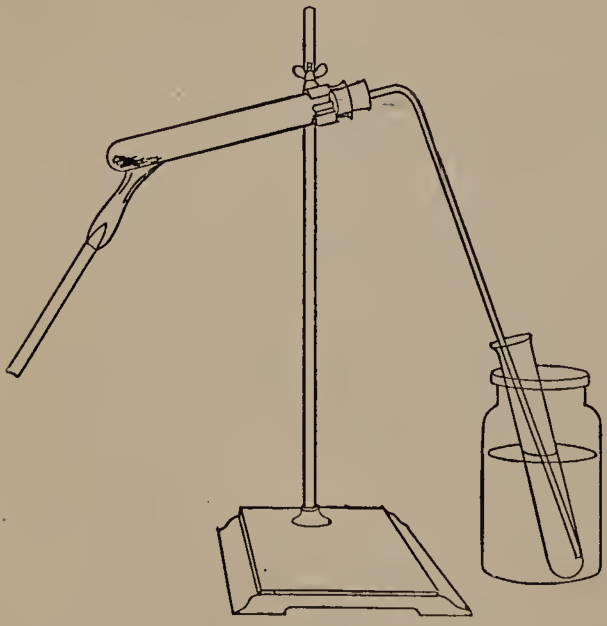

Fig. 91. Preparation of bromine.

Heat the mixture very gently but not enough to boil the black liquid through the delivery tube. Stop heating when all the red vapor is driven over.

b. Properties. *Note the color and odor* (***cautiously***) *of bromine vapor and the color of the liquid bromine.*

Fill the test tube containing the bromine about two-thirds full of water. Set the mixture aside.

Is bromine heavier or lighter than water? Is it soluble in water?

Pour about 2–3 cc. of the "bromine water" just prepared into a test tube and add not more than 1 cc. of carbon disulfide. Cover the tube with the thumb and shake the contents vigorously. Most of the carbon disulfide settles to the bottom. *Compare the colors of the disulfide and water and so the relative solubility of bromine in these two liquids.*

c. Test for a bromide. Dissolve a small crystal of potassium (or sodium) bromide in about 5 cc. of water. *Note the color of the solution.* Add 2 cc. of carbon disulfide and shake the mixture. *Why is the carbon disulfide not colored by the bromine?*

Orange brown

Now add a few drops of freshly prepared chlorine water and shake the contents vigorously. Allow the mixture to settle. *What substance has been set free by the chlorine? Complete the equation*

$$KBr + Cl_2 \longrightarrow \underline{\hspace{6em}} + \underline{\hspace{6em}}$$

d. Summarize the properties of bromine: *color, odor, density of vapor* (compared with air), *density of liquid* (compared with water), *relative solubility in water and carbon disulfide,* and *activity compared with chlorine.*

EXPERIMENT 61

Iodine — Preparation and Properties

Apparatus. — Test tubes and rack. Bunsen burner. Evaporating dish. Tripod. Crystallizing dish. Asbestos gauze. Mortar and pestle.

Materials. — Potassium iodide. Manganese dioxide. Concentrated sulfuric acid. Alcohol. Potassium iodide solution. Carbon disulfide. Chlorine water. Bromine water. Sodium thiosulfate solution.

a. Preparation. Pour into an evaporating dish about 3 cc. of water and add about twice that volume of concentrated sulfuric acid. Pulverize several crystals (about 2 g.) of potassium iodide with twice as much manganese dioxide and add the mixture to the acid. Set the evaporating dish on the asbestos gauze supported on the tripod and place a crystallizing dish half full of cold water upon it in such a way that the iodine vapor may be condensed on the bottom of the cool dish (Fig. 92).

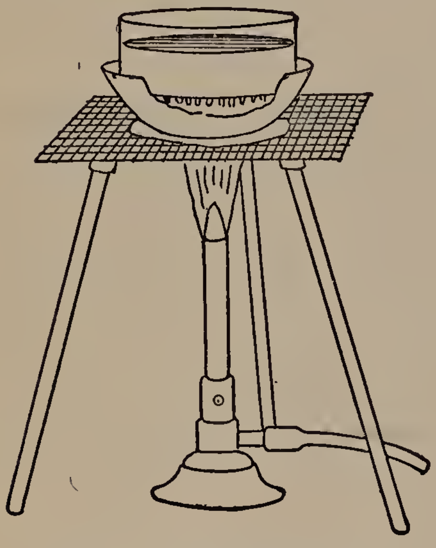

Fig. 92. Preparation of iodine.

Warm the evaporating dish with a very small flame. If the iodine vapor (violet) escapes, decrease the heat and refill the crystallizing dish with cold water. The iodine vapor condenses as a solid (*i.e.* sublimes) on the bottom of the cold dish. Scrape off the iodine crystals on a paper.

b. Properties. — *Solvents.* Try to dissolve a crystal of iodine in water by shaking it with a few cubic centimeters of water. *Is the iodine quite insoluble in water?*

To the test tube containing water and iodine, add an equal volume of carbon disulfide and shake the mixture. *Compare the solubility of iodine in water and in carbon disulfide.*

Try the solubility of iodine in ethyl alcohol; also in solutions of potassium iodide and sodium thiosulfate ("hypo"). Tabulate the solubilities of iodine in these various solvents and the colors of the solutions as follows:

SOLVENT	COLOR OF SOLUTION	SOLUBILITY
Water Carbon disulfide, etc.		Very great, moderate or slight

c. Replacement of iodine. Dissolve a small crystal of potassium iodide in about 5 cc. of water and then add a few cubic centimeters of carbon disulfide. Shake the mixture. *Why is the liquid not colored with the iodine?*

Add a few drops of chlorine water and shake the liquids vigorously. Allow the mixture to settle. *What substance has been set free by the chlorine? Complete the equation.*

$$KI + Cl_2 \longrightarrow \underline{\qquad\qquad} + \underline{\qquad\qquad}$$

To another portion of potassium iodide solution, add bromine water and carbon disulfide. After shaking the mixture let it settle. *What substance has been set free by the bromine? Complete the equation:*

$$KI + Br_2 \longrightarrow \underline{\qquad\qquad} + \underline{\qquad\qquad}$$

Assuming that the more active element replaces the less active element, which of these halogens is the most active and which is the least active?

d. Summarize the properties of iodine: *color of solid, color of vapor, density of vapor* (compared with air), *density of solid* (compared with water), and *relative solubilities in the various solvents used.*

EXPERIMENT 62

The Hydrogen Halides

Apparatus. — Test tubes and rack. Bunsen burner.

Materials. — Sodium chloride. Sodium or potassium bromide. Sodium or potassium iodide. Concentrated sulfuric acid. Blue litmus paper. Concentrated ammonium hydroxide. Filter papers.

a. Hydrogen chloride. Put about 1 g. of common salt (sodium chloride, NaCl) in a test tube and add a few drops of concentrated sulfuric acid. If no action is visible, add a few drops more of sulfuric acid and warm the tube gently. *What happens?*

Blow across the mouth of the tube. *What is the result?* Hold a strip of moistened blue litmus paper near the mouth of the tube. *Result?* Hold a piece of filter paper which has been moistened in ammonia water near the tube. *Explain the formation of a white cloud.*

Write the equation for the preparation of hydrogen chloride.

b. Hydrogen bromide. Repeat in every detail the experiment (a) using potassium bromide. *Answer the same questions.* Remember that all the hydrogen halides are colorless. *How do you account for the colored gas in the test tube? What element gives this color? What does it indicate as to the stability of hydrogen bromide? What gas is formed besides hydrogen bromide?* (Smell the gas very cautiously.)

c. Hydrogen iodide. Again repeat the experiment as in part (a) using potassium iodide. *Answer the same questions.* The amount of the fuming when you blow across the mouth of the test tube indicates the quantity of hydrogen iodide coming from the tube.

What is the colored vapor?

What gas, which you can recognize by its odor, is formed besides hydrogen iodide?

d. Comparison. *What does this experiment show with regard to the relative stability of hydrogen chloride, hydrogen bromide, and hydrogen iodide?*

Why are hydrobromic and hydriodic acids not usually prepared in this way?

EXPERIMENT 63

Hypochlorous Acid — Bleaching

Apparatus. — Test tubes and rack. Carbon dioxide generator (Exp. 12). Funnel.

Materials. — Bleaching powder (chloride of lime). Filter papers. Marble chips. Concentrated hydrochloric acid. Colored calico. Litmus paper.

a. Preparation. Make a saturated solution of bleaching powder (chloride of lime) by adding about 2 cc. of the powder to 20 cc. of water in a test tube and by shaking occasionally. Filter off the clear solution and pass carbon dioxide through the liquid for at least 5 minutes. The equation for this reaction is as follows:

$$CaOCl_2 + H_2CO_3 \longrightarrow CaCO_3 \downarrow + HOCl + HCl$$

By filtering off the precipitate, calcium carbonate, we obtain a clear solution containing the hypochlorous acid.

b. Use in bleaching. Place in the solution of the hypochlorous acid, just prepared, small strips of (1) litmus paper, (2) paper with printing on it, (3) paper on which there is writing in ink, (4) paper with pencil marks, and (5) colored calico. *Record the effect on each. Which of these materials is colored with organic compounds and which with free carbon? Does hypochlorous acid oxidize free carbon? What does it oxidize?*

Optional work. Unbleached cotton cloth may be bleached with chloride of lime as follows: — (1) First clean the cloth of grease by boiling the cloth in a weak solution of washing soda (5% Na_2CO_3 solution). (2) Then wash and place the cloth in a bleach bath (10% $CaOCl_2$ solution). (3) After 5 minutes partially wring and place the cloth in a very dilute solution of sulfuric acid (5 cc. conc. H_2SO_4 in 100 cc. water). (4) Again place the cloth in the bleach solution and thus alternate until the color is removed. (5) To remove any traces of the hypochlorous acid left in the cloth, wash it several times in a solution of sodium sulfite (5% Na_2SO_3 sol.). Mount in your notebook a sample of the original cloth and another as bleached.

EXPERIMENT 64

Hydrogen Peroxide

Apparatus. — Small flask. Stirring rod. Glass jar. Funnel. Test tubes and rack.

Materials. — Sodium peroxide. Cracked ice. Litmus papers. Dilute hydrochloric acid. Powdered manganese dioxide. Wooden splint. Lead nitrate solution. Ammonium sulfide solution. Potassium dichromate solution. Dilute sulfuric acid. Ether.

a. Preparation. Dissolve in a flask about 1 cc. of sodium peroxide in 100 cc. of ice-cold water. Add the sodium peroxide to the water in very small quantities with constant stirring and cooling in running water. Test a drop of the solution with litmus paper. Now add a few drops of hydrochloric acid and shake and cool the flask. Continue this process until the solution is slightly acid. *What are the products of this reaction? Write the equation.*

b. Properties. ***Stability.*** Take half a test-tubeful of the solution and have ready on a piece of paper about 1 cc. of finely powdered manganese dioxide. Light a wooden splint and then put the manganese dioxide into the solution. Test the gas in the test tube for oxygen with the glowing splint. The manganese dioxide is unchanged and serves as a catalyst. *Is hydrogen peroxide stable or not?*

Oxidizing agent. Precipitate some lead sulfide by adding a few drops of ammonium sulfide solution ($(NH_4)_2S$) to a few cubic centimeters of lead nitrate solution ($Pb(NO_3)_2$). Collect the precipitate on a filter paper and wash with water 2–3 times. Then pour over the precipitate some of the hydrogen peroxide solution. *What lead compound is formed? Write the equation.*

c. Test for hydrogen peroxide. Nearly fill a large test tube with water and add just enough potassium dichromate solution to tinge it a light yellow. Now add a couple of drops of dilute sulfuric acid to set free dichromic acid. Take a portion of hydrogen peroxide solution in a test tube and add 2–3 cc. of ether.

CAUTION. **Have no flames near when using ether.**

Close the tube with the thumb and shake the liquids. Add one drop of dichromic acid solution and shake the liquids again. If in doubt about the result, add another drop of the dichromic acid solution. This is one of the most characteristic and delicate tests for hydrogen peroxide.

Optional work. Repeat this experiment using instead of sodium peroxide **lead dioxide**, **barium dioxide**, and powdered **manganese dioxide**. *Is hydrogen peroxide formed in each case? What are the differences in behavior and constitution between a true peroxide and other oxides?*

EXPERIMENT 65

ACIDS OF PHOSPHORUS

Apparatus. — Wide-mouth bottle. Glass square. Deflagrating spoon. Beaker. Tripod. Asbestos gauze. Bunsen burner. Test tubes and rack.

Materials. — White phosphorus. Dilute nitric acid. Dilute ammonium hydroxide. Silver nitrate solution. Ammonium molybdate solution. Litmus papers.

CAUTION. **White phosphorus takes fire readily. It should never be handled with the hands.**

a. Preparation. Place about 20 cc. of distilled water in a clean wide-mouth bottle or pint fruit jar. Line a deflagrating spoon with asbestos paper and put in it a piece of white phosphorus. Ignite it by touching it with a hot wire and lower the burning phosphorus into the bottle or jar, quickly covering the mouth of the bottle with a glass plate (Fig. 93). As soon as the phosphorus stops burning, remove the spoon and shake the bottle to dissolve the phosphorus pentoxide (P_2O_5). *Test the solution with litmus paper. Write the equations.*

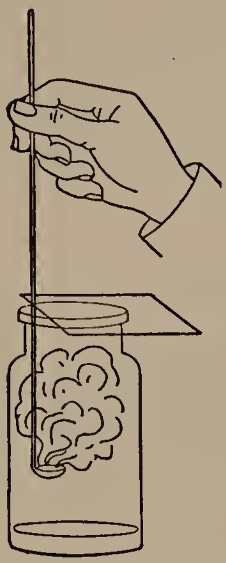

Fig. 93. Burning phosphorus on a deflagrating spoon in a bottle with a little water.

Pour 2–3 cc. of dilute nitric acid into the solution in a beaker and boil it down to about half its original volume. The hydrogen ion of the nitric acid acts as a catalyst to convert the metaphosphoric acid (HPO_3) by addition of water to orthophosphoric acid (H_3PO_4). *Write the equation.*

b. Tests. Add to a portion of the orthophosphoric acid nearly enough dilute ammonium hydroxide to neutralize the acid; then add silver nitrate solution drop by drop until a precipitate is formed. *Name and describe this precipitate. Write the equation.*

To about 10 cc. of a solution of ammonium molybdate add a few drops of the orthophosphoric acid. Let the mixture stand for several minutes and then warm gently. A precipitate shows the presence of the phosphate ion (PO_4^{---}). *Describe the precipitate.*

Try the effect of adding ammonium hydroxide solution until the solution is alkaline. *Note the result.* Again acidify with nitric acid and *record the result.*

Optional work. In an evaporating dish put 10 g. of powdered phosphate rock (apatite) and add 4 cc. of water and then 4 cc. of concentrated sulfuric acid. Stir and warm the mixture for 10 minutes. Add about 10 cc. more of water and filter. Test the filtrate for a phosphate. *Has the phosphate rock been converted into a soluble phosphate by the action of sulfuric acid?*

Test a commercial superphosphate fertilizer for a soluble phosphate.

EXPERIMENT 66

SOIL TESTING

Apparatus. — Beaker. 3 pint fruit jars. Graduated cylinder. Magnifying glass.
Material. — Sample of soil (from home garden). Litmus papers.

a. Litmus test for acidity. Place some soil in a beaker or tumbler and moisten it well with distilled water. Put a strip of blue litmus paper on the moist mass. If the soil is acid, the litmus paper will turn pink within 15 minutes.

Sometimes it is necessary to test soil for acidity where distilled water is not available. In this case the test may be made by taking a handful of moist soil direct from the field and pressing it into a ball. Then break the ball in two so that the litmus paper may be placed between the two halves. After 10 minutes examine the litmus paper.

NOTE. It is well to handle the litmus paper with forceps because the perspiration of the hands is usually slightly acid. Use only the best litmus paper.

What are the means of correcting acidity in the soil?

b. Per cent of sand, silt, and clay. Put about 2 tablespoonfuls of fine soil in a pint fruit jar three-fourths full of water. Shake the mixture vigorously for several minutes and then let it stand for one minute. Pour off the muddy water into a second jar. The sediment remaining in the first jar is composed mainly of *sand.*

When the second jar has been standing 5 minutes pour off the muddy water into a third jar. Add more water to this jar and shake it vigorously. After it has stood for at least 2–3 hours, pour off the water. The residue left in the second jar was largely *silt;* the residue in the third jar is almost entirely *clay.* Dry each of these three residues and determine the volumes of each portion in a graduated cylinder. *Compute the* *per cent of sand, silt, and clay in the soil. Note how fine the particles of each ingredient are and examine them under a magnifying glass.*

Optional work. It is possible to determine approximately the degree of acidity of soil by the **Truog test.** Place 10 g. of soil in a flask with 95 cc. of distilled water. Prepare a mixture of zinc sulfide and calcium chloride by dissolving 50 g. of neutral calcium chloride in 250 cc. of water and then adding 5 g. of finely powdered zinc sulfide. Add 5 cc. of this zinc sulfide-calcium chloride mixture to the mixture of soil and water. Heat the mixture to boiling and after it has boiled for 1 minute place a strip of lead acetate paper * over the mouth of the flask and continue boiling for 2 minutes longer. The darkening of the acetate paper shows the presence of acid in the soil and the degree of acidity is roughly indicated by the depth of color. A convenient portable outfit is shown in figure 94; this makes use of a small alcohol heater to boil the water and a chart of standard colors is furnished.

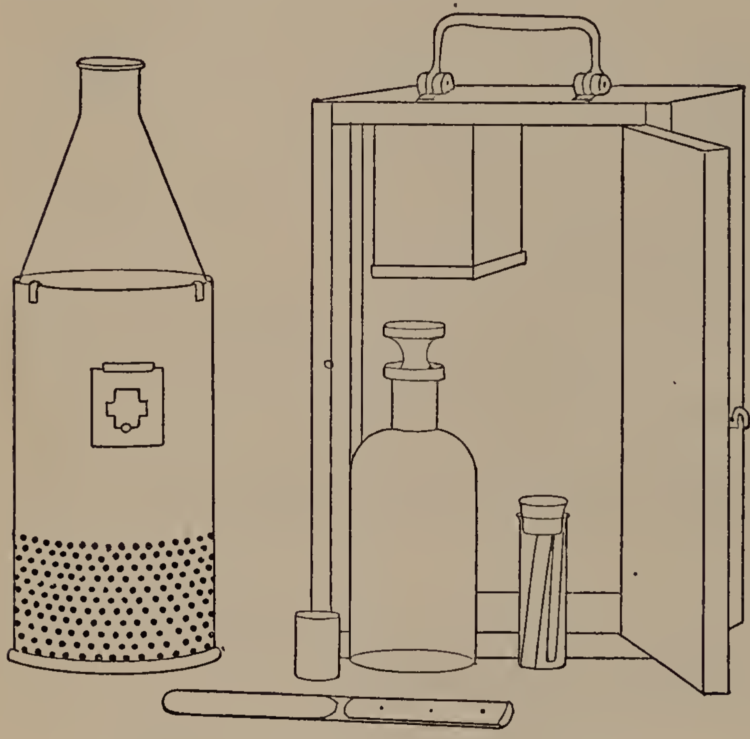

Fig. 94. Truog soil testing outfit.

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* The lead acetate paper is prepared by dipping a piece of white filter paper into a 10% solution of lead acetate and then drying the paper on a pane of glass.

EXPERIMENT 67

Compounds of Silicon

Apparatus. — Mortar and pestle. Iron crucible. Pipe-stem triangle. Tripod. Bunsen burner. Funnel. Evaporating dish. Asbestos gauze.

Materials. — Clean sand (silica). Sodium carbonate (powdered). Filter papers. Dilute hydrochloric acid.

a. Sodium silicate. Thoroughly mix 1 g. of clean sand (SiO_2) with 5–6 g. of dry sodium carbonate by grinding them together in a mortar. Put the mixture into an iron crucible and heat gently over a Bunsen flame until the effervescence ceases and then to a red heat until the whole mass is fused. Allow the fused mass to become thoroughly cold and then dissolve as much of it as is soluble in 25 cc. of boiling water. Filter the mixture and discard the residue. Concentrate the filtrate by evaporating the solution to about one-third its original volume. This solution contains sodium silicate or water-glass.

b. Silicic acid. Acidify the solution of sodium silicate by the addition of dilute hydrochloric acid. The gelatinous precipitate is silicic acid (H_2SiO_3). Slowly evaporate the acid mixture, heating it gently to avoid sputtering, until a dry white powder remains. Heat almost to redness for a few minutes and then let it cool.

Extract the sodium chloride from the residue by adding 25 cc. of water and boiling gently for several minutes. Identify what remains.

What was formed during the fusion? Write the equation.

What was formed by adding hydrochloric acid? Write the equation.

What was the object of heating nearly to redness? Write the equation.

Optional work. Mix thoroughly in a beaker about 30 cc. of the commercial solution of water glass with about twice its volume of water. Drop a small crystal of each of the following substances into this solution: copper sulfate, nickel sulfate, cobalt nitrate, zinc sulfate, and ferrous sulfate. Let the solution stand quietly over night. This is sometimes called "**The Chemist's Garden.**" Record and explain the changes observed.

EXPERIMENT 68

BORAX BEAD TESTS FOR METALS

Apparatus. — Platinum (mounted). Bunsen burner.
Materials. — Borax (powdered). Cobalt nitrate. Manganese dioxide. Chromium sulfate. Ferrous sulfate. Nickel nitrate. Unknowns.

a. Preliminary. Ordinary borax is hydrated sodium tetraborate ($Na_2B_4O_7 \cdot 10\,H_2O$). When heated, borax swells due to the evaporation of the water of crystallization and then melts to a clear glassy mass. The oxides of certain metals impart characteristic colors to beads of borax which acts as a flux, dissolving the metallic oxide.

b. Borax beads. Attach one end of a piece of platinum wire to a piece of glass tubing by melting the glass. Bend the other end of the platinum wire into a loop, about 3 mm. in diameter. Heat the wire in the Bunsen flame and while still hot dip it quickly in some powdered borax. Heat strongly in the flame. After a moment the borax shrinks into a small transparent bead. Dip this again into the borax and repeat this process until you have formed a round bead as large as the loop.

c. Tests for metals. Touch this borax bead, while still hot, to a tiny bit of some cobalt compound, such as cobalt nitrate. Heat again in the hot outer portion (Fig. 95) of the Bunsen flame (oxidizing), until the color is uniform. If the bead is black, too much of the salt was used. Try again. To remove the bead dip it, while red hot, in water. *Record the characteristic color in a table.*

METALS	FORMULA OF COMPOUND	COLOR OF BEAD IN OXIDIZING FLAME
Cobalt, etc.		

Repeat this experiment with compounds of **manganese**, **chromium**, **iron**, and **nickel**, using each time a newly made bead. Learn the characteristic colors of these metals.

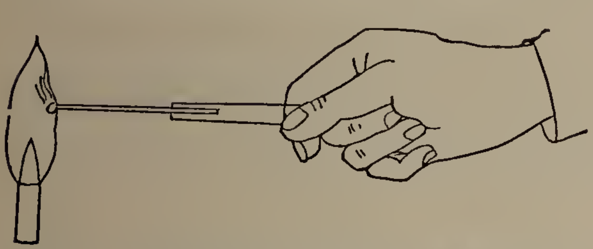

Fig. 95. Making a borax bead in the outer flame.

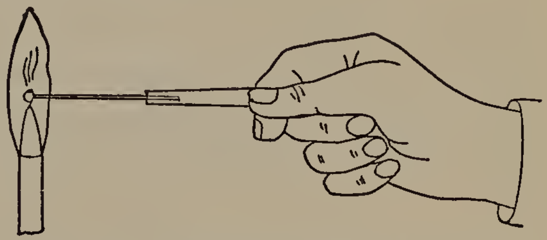

Fig. 96. Making a borax bead in the inner flame.

Ask the instructor for an unknown salt to test by the bead method.

Optional work. Repeat this experiment using the **reducing flame**, *i.e.* just at the tip of the inner cone (Fig. 96), with each metal. Also note any differences in color between the bead when hot and when cold.

EXPERIMENT 69

ALUMINUM HYDROXIDE — PREPARATION AND USES

Apparatus. — Test tubes and rack. Wide-mouth bottle or glass cylinder.

Materials. — Aluminum sulfate. Limewater. Clay. Cochineal or indigo. Dilute ammonium hydroxide.

a. Preparation. Prepare about 5 cc. of aluminum sulfate solution ($Al_2(SO_4)_3$) or the same quantity of any aluminum salt and add limewater until a precipitate is formed. *Describe the appearance of the precipitate* ($Al(OH)_3$) *and write the equation.*

b. Clarifying muddy water. Fill a glass bottle or cylinder about two-thirds full of water and render the water turbid with a little finely pulverized clay. Pour in about 10 cc. of aluminum sulfate solution and mix it thoroughly with the turbid water. Then add slowly, without stirring, 20 cc. of limewater. Set the cylinder aside to settle. *What precipitate was formed in the turbid water? Explain how the suspended particles of clay are removed.*

c. Mordant. Take about 5 cc. of water in a test tube and add enough cochineal or indigo solution to color the water faintly. Add an equal volume of aluminum sulfate solution and enough ammonium hydroxide to precipitate the aluminum hydroxide. Shake the test tube and allow the contents to settle. *Note the effect on the color of the solution.*

NOTE. The particles of dyestuff are much smaller than those of the clay, but the aluminum hydroxide collects them. In dyeing cloth aluminum hydroxide is sometimes used as a **binder** (mordant) between the cloth and the dye.

Optional work. In dyeing cloth it is necessary first to remove the sizing by boiling the cloth in very dilute hydrochloric acid (about 1%); then rinse the goods in water containing a little ammonium hydroxide to neutralize any traces of acid and rinse again in water. Prepare in this way several squares of white cotton cloth.

Boil one piece of cloth for 2 minutes in a solution of aluminum sulfate. Then wring the cloth and place it in ammonium hydroxide solution and shake it for 2 minutes. Wring the cloth again. It is now mordanted with aluminum hydroxide.

In an evaporating dish put 50 cc. of water and 5 cc. of alizarin paste. Place in this dye a piece of unmordanted cloth and a piece of mordanted cotton and boil for 10 minutes. Wash the pieces of cloth in running water and dry on a pane of glass. Paste each in your notebook with a descriptive label.

EXPERIMENT 70

TREATMENT OF HARD WATER

Apparatus. — Carbon dioxide generator (Exp. 12). Large test tube. Test tubes and rack. 2 small beakers. Funnel. Bunsen burner.

Materials. — Limewater. Marble chips. Concentrated hydrochloric acid. Calcium sulfate solution. Magnesium sulfate solution. Sodium carbonate solution. Filter papers. Concentrated ammonium hydroxide. Ammonium oxalate solution. Ammonium chloride solution. Solution of di-sodium phosphate.

a. Temporary hardness. Dilute about 10 cc. of saturated limewater by adding an equal volume of water. Pass a steady stream of carbon dioxide through the half-saturated limewater until the precipitate ($CaCO_3$) is formed and redissolved. The clear liquid contains calcium bicarbonate ($CaH_2(CO_3)_2$) and is called water of temporary hardness. *Write two equations to represent these changes.*

b. Removing temporary hardness. To about 5 cc. of the hard water just prepared, add about 3 cc. of saturated limewater and shake the mixture. *What is the precipitate? Write the equation.*

Boil for several minutes about 5 cc. of the hard water prepared in (**a**). The acid salt (bicarbonate) is changed back into the normal salt with the loss of carbonic acid. Set the tube with contents aside to settle. *Write the equation. Compare the amounts of precipitated calcium carbonate formed by boiling and adding limewater. Explain.*

c. Permanent hardness. Pour 10 cc. of a solution of calcium sulfate * into a small beaker and the same volume of magnesium sulfate solution into another beaker. Add to each 5 cc. of soidum carbonate solution. (Compare solubility of calcium

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* The calcium sulfate solution should be prepared some time beforehand. Shake some plaster of Paris with water, let it stand for some time and then filter.

carbonate and sulfate. Table in Appendix.) Filter each solution. *What is the precipitate in each case? Write the equations.*

Add to the filtrate from the calcium sulfate solution 5 cc. of concentrated ammonium hydroxide and 5 cc. of a solution of ammonium oxalate. If the calcium ion (Ca^{++}) is present, a white precipitate of calcium oxalate will be formed.

Add to the filtrate from the magnesium sulfate solution 5 cc. of a solution of ammonium chloride, 5 cc. of concentrated ammonium hydroxide, and 5 cc. of a solution of di-sodium phosphate. If the magnesium ion (Mg^{++}) is present, a white precipitate of magnesium ammonium phosphate will be formed.

What does this experiment show in regard to "softening" permanent hard waters with sodium carbonate?

Optional work. Test the temporary and permanent hard waters with soap solution to determine the readiness with which they form permanent soap suds. Also test with the soap solution the hard waters, after they have been softened. *What is the objection to calcium soap? Why are magnesium salts particularly bad in waters to be used in boilers?*

EXPERIMENT 71

MORTAR AND CONCRETE

Apparatus. — Asbestos gauze. Tripod. Bunsen burner. Evaporating dish. Test tubes. 2 wide-mouth bottles with corks.

Materials. — Marble chips. Lumps of quicklime. Litmus papers. Sand (clean, sharp). Dilute hydrochloric acid. Portland cement.

a. Making quicklime. Place a small chip of marble (about 5 mm.) on a corner of the wire gauze and heat it as hot as possible for 15–20 minutes. The product is calcium oxide or quicklime (CaO). *Compare it with marble. Write the equation for this action.*

b. Slaking quicklime. Pour a little boiling water on a lump of quicklime (about 25 g.) in an evaporating dish. Pour water upon it a little at a time as long as it is taken up *readily*. Do not add too much water. Watch the lump for some time, and note any changes (heat, volume, appearance). At the end you should have a smooth, stiff paste. *Test with litmus and write the equation for this process.*

c. Mixing mortar. Mix with the paste just prepared about 50 g. of sand and add more water if required. Make up part of the mortar into a ball and place in a wide-mouth bottle. Cork it tightly. Spread the rest on a board. After two days compare the two samples of mortar. Test each with dilute hydrochloric acid. *Explain the difference in the two samples of mortar. Write the equation for the setting of mortar.*

d. Portland cement. Mix a tablespoonful of cement with twice its bulk of clean sharp sand and then add slowly enough water to make a stiff paste. Take part of this mixture and make a small ball. Place this in a wide-mouth bottle and cork it tightly. Pour the rest of the paste into a small pasteboard or wooden box which has been greased. After two days examine each sample of concrete. *What advantages has concrete over mortar as a building material?*

EXPERIMENT 72

FERROUS COMPOUNDS

Apparatus. — Test tubes and rack. Bunsen burner. Funnel. Small flask and cork. Tripod. Asbestos gauze.

Materials. — Small iron nails (or steel wool). Dilute hydrochloric acid. Filter papers. Dilute ammonium hydroxide. Potassium ferrocyanide solution. Potassium ferricyanide solution. Potassium sulphocyanate solution.

a. Making ferrous chloride. Place in a test tube about a spoonful of small iron nails or a small bundle of steel wool and add just enough dilute hydrochloric acid to dissolve the iron. Warm the acid to keep the action going briskly. *Identify the* *gas given off. Account for its odor, and for the insoluble black residue.*

Filter the liquid into a small flask containing about 50 cc. of boiling water. Add a few nails and cork the flask loosely, removing the cork only when pouring out some of the solution. *Note the color of the solution. Write the equation for the formation of ferrous chloride* ($FeCl_2$).

b. Ferrous hydroxide. Pour about 5 cc. of this ferrous chloride solution into a test tube and add dilute ammonium hydroxide until the solution after shaking gives the odor of ammonia. *What is the precipitate? Write the equation for its formation.*

Filter and open out the paper with the precipitate. Lay it aside to dry. *Note the changes in the color of the precipitate when exposed to the air.*

c. Tests. To about 5 cc. of ferrous chloride solution add about 1 cc. of potassium ferrocyanide ($K_4Fe(CN)_6$) solution. Note that the white precipitate immediately begins to turn blue. *Write the equation, assuming that potassium ferrocyanide ionizes as follows;*

$$K_4Fe(CN)_6 \longrightarrow 4\ K^+ + Fe(CN)_6^{----}$$

To another portion of ferrous chloride solution add potassium ferricyanide ($K_3Fe(CN)_6$) solution. *Note the color produced and whether there is a precipitate or simply a colored solution.* To determine the color of a very deeply colored solution, dilute with much water.

To still another portion of ferrous chloride solution add potassium sulphocyanate (KSCN) solution. The result should be negative.

Give one distinctive test for ferrous salts (Fe^{++}).

NOTE. Keep the rest of the ferrous chloride solution for Exp. 73.

EXPERIMENT 73

Ferric Compounds

Apparatus. — Small flask. Tripod. Asbestos gauze. Bunsen burner. 1-hole rubber stopper with Bunsen valve (Fig. 96). Test tubes and rack.

Materials. — Ferrous chloride solution (from Exp. 72). Concentrate hydrochloric and nitric acids. Concentrated ammonium hydroxide. Solutions of potassium ferrocyanide, potassium ferricyanide, and potassium sulphocyanate. Steel wool. Iron turnings.

a. Ferrous to ferric. Boil the remainder of the ferrous chloride solution (Exp. 72) with a little concentrated hydrochloric acid and nitric acid for about 2 minutes. *Note the change in color* — this is characteristic of ferric solutions. The nitric acid serves as an oxidizing agent. Complete the equation:

$$FeCl_2 + HCl + [O] \longrightarrow FeCl_3 + \underline{\qquad\qquad}$$

b. Ferric hydroxide. To a portion of the ferric chloride solution, add an excess of ammonium hydroxide. If the precipitate is green, the above action (called oxidation) was incomplete and should be repeated. *Write the equation for the formation of ferric hydroxide* ($Fe(OH)_3$). *Explain the change in color in the ferrous hydroxide prepared in Exp. 72.*

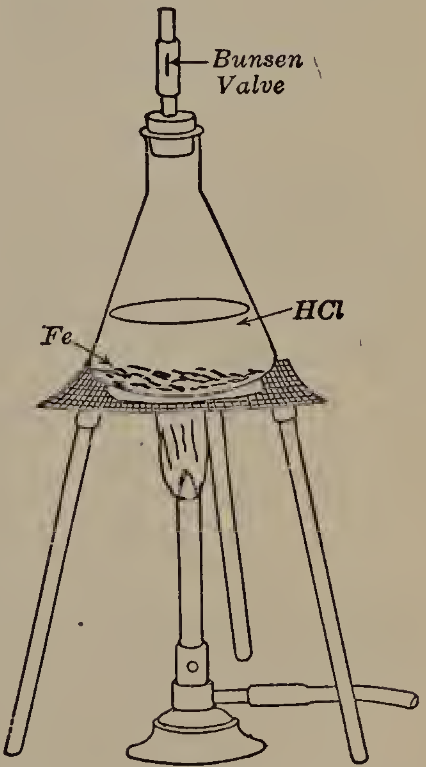

Fig. 97. Flask with Bunsen valve.

c. Tests. Repeat all the tests of Exp. 72 (c) using ferric chloride solution instead of ferrous chloride. *Give two distinctive tests for a ferric salt.*

d. Reduction of a ferric compound. Boil in a small flask * (Fig. 97) the remainder of the ferric chloride solution with a few drops of concentrated hydrochloric acid and some iron turnings or small iron nails. Keep the action going vigorously until the characteristic color of ferric compounds has disappeared. Pour off some of the clear solution and test quickly for the ferrous ion. *Complete the equation:*

$$FeCl_3 + \quad Fe \longrightarrow \quad FeCl_2$$

e. Summarize the results of the following reagents on ferrous and ferric compounds in tabular form:

REAGENTS	FERROUS	FERRIC
Ammonium hydroxide		
Potassium ferrocyanide		
Potassium ferricyanide		
Potassium sulphocyanate		

Optional work. Try the action of hydrogen sulfide and of ammonium sulfide on ferric chloride solution. *Explain the results.* Determine what happens when a solution of sodium carbonate is added to a solution of a ferric salt.

EXPERIMENT 74

COBALT NITRATE TESTS FOR METALS

Apparatus. — Forceps. Blowpipe. Bunsen burner.

Materials. — Charcoal block (or block of plaster of Paris). Zinc sulfate. Solution of cobalt nitrate 5%. Aluminum sulfate. Magnesium carbonate. Unknowns.

a. Zinc. Make a cavity in a charcoal block with the top of the forceps. Place a little of some zinc compound, such as

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* It is well in preparing ferrous compounds to provide the flask with a Bunsen valve. This is a short rubber tube with a slit cut in the side and plugged at the end.

zinc sulfate, in this cavity and heat it as hot as possible at the tip of a small blowpipe flame (Fig. 98). *After the residue has cooled, record its color.*

Put a drop or two of cobalt nitrate solution on the residue and reheat it intensely. *After it has cooled, record the color of the mass that remains on the charcoal block.*

b. Aluminum. Make a fresh cavity in the block and place in it some aluminum compound, as aluminum sulfate, and repeat the test made with the zinc compound. *Record the characteristic coloration which is obtained.*

c. Magnesium. Repeat (a), using some compound of magnesium, such as magnesium carbonate or sulfate. Heat the mass until it glows brightly. Cool and moisten the fused mass with only a very little cobalt nitrate solution. Reheat intensely and after the mass has cooled, note carefully the delicate coloration.

Fig. 98. Using a blowpipe flame on charcoal.

d. Unknown. Obtain from the instructor some compound of aluminum, zinc, or magnesium and identify the metal with the blowpipe and cobalt nitrate solution.

EXPERIMENT 75

REPLACEMENT OF METALS

Apparatus. — Test tubes and rack.

Materials. — Five strips of zinc (10 × 1 cm.). Sandpaper. Solutions of copper sulfate, lead nitrate, silver nitrate, zinc nitrate, and mercurous nitrate. Dilute sulfuric acid. 3 strips of lead. 4 strips of copper.

a. Zinc. Prepare 5 strips of zinc (10 × 1 cm.) and clean each strip with sandpaper. In separate test tubes place about

5 cc. of solutions of lead nitrate, copper sulfate, silver nitrate, mercurous nitrate, and of dilute sulfuric acid. Label each tube with the formula of the substance it contains and also with the symbol of the positive ion in each case. Place a strip of zinc in each solution in such a way that the top of the strip bends over the edge of tube (Fig. 99). After 5–10 minutes withdraw the strips and examine them minutely. Remember that finely divided metals present a very different appearance from the same metals in compact polished condition. Tabulate your results as follows:

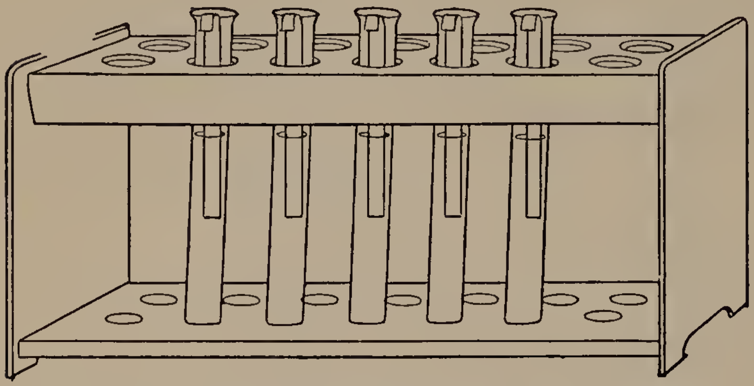

Fig. 99. Strips of metal partly immersed in solutions.

Metal Strip	Solution	Deposit
Zinc etc.	Lead nitrate	Lead

b. Lead. Prepare 3 strips of bright clean sheet lead. Place in separate test tubes about 5 cc. each of solutions of mercurous nitrate, silver nitrate, and copper sulfate. Label each tube as in (a) and place a strip of lead in each solution. *Examine later and tabulate the results.*

c. Copper. In the same way prepare 4 strips of copper and place them in another set of test tubes containing solutions of lead nitrate, silver nitrate, zinc nitrate, and mercurous nitrate. *Note any change in the color of the solution left in the test tube. Record negative as well as positive results. For each case giving a positive result write the equation showing the ions. In those cases which give a negative result, explain why it is so.* Compare your results with the Replacement Series of Metals on p. 420 in B. & C.

Optional work. Study the action of dilute sulfuric acid on the strips of zinc which have been partly coated with another metal (**a**) and on a clean strip of zinc. *Do you observe any difference in the action?* A metal coated with another metal is called a "couple." *Which metal of the couple is attacked by the acid — the more active or the less active one?* Galvanized iron and tin plate are examples of couples. *Explain why tinned iron is more apt to rust than galvanized iron.*

EXPERIMENT 76

Copper Compounds

Apparatus. — Test tubes and rack. Small flask. Tripod. Asbestos gauze. Bunsen burner.

Materials. — Crystals of copper sulfate, sodium chloride, copper chloride, and sodium sulfate. Copper filings. Concentrated hydrochloric acid. Sodium hydroxide solution. Litmus papers. Potassium ferrocyanide solution. Concentrated ammonium hydroxide.

a. Color of cupric ions. Dissolve in separate test tubes small crystals of copper sulfate, copper chloride, sodium chloride, and sodium sulfate in 15 cc. of water. *Note the color of the solutions. What are the colors, respectively, of the cupric, sodium, sulfate, and chloride ions?*

b. Cuprous compounds. Dissolve a teaspoonful of cupric chloride in 15 cc. of water in a small flask. Add a teaspoonful of copper filings and 5 cc. of concentrated hydrochloric acid and boil gently until the green color has quite disappeared. Pour half of the product into 100 cc. of water. *What is the color of the cuprous ions? Write the equation for the reduction of cupric salts with copper.*

To the remainder of the cuprous chloride solution add sodium hydroxide solution until the solution is strongly alkaline. *Note the color of the precipitate.* Try the effect of heating it. *Write the equations representing these changes.*

c. Cupric compounds. Test a solution of copper sulfate with litmus paper. Explain the reaction with the ionic theory. (See § 301 in B. & C.)

Add sodium hydroxide solution to a cold solution of copper sulfate as long as it forms a precipitate. *Note the color of the precipitate.* Heat the mixture until no further change takes place. *Write the equations for these changes.*

d. Tests for cupric ions. Add a few drops of copper sulfate solution to half a test-tubeful of water and then add potassium ferrocyanide solution. The precipitate is cupric ferrocyanide ($Cu_2Fe(CN)_6$).

Another characteristic test for the cupric ion is to add concentrated ammonium hydroxide drop by drop to the copper solution until no further change is produced. The deep blue color is due to a complex copper-ammonia ion ($Cu(NH_3)_4^{++}$).

Optional work. Find out how copper sulfate is used as an insecticide (Paris green) and as a fungicide (Bordeaux mixture). Consult books on horticulture and various Farmers' Bulletins published by the U. S. Department of Agriculture for detailed directions.

EXPERIMENT 77

SILVER COMPOUNDS IN PHOTOGRAPHY

Apparatus. — Test tubes and rack. Funnel. Shallow dish or tray.

Materials. — Silver nitrate solution. Potassium bromide solution. Filter papers. Tube of photographic developing powder. Sodium thiosulfate (hypo) solution. Potassium chloride solution.

a. Preliminary. The sensitive plate or film is coated with silver bromide (AgBr) embedded in gelatin. After exposure to light (the eye detects no changes), the plate is placed in a developing solution. This solution contains various carbon compounds derived from benzene, such as pyrogallic acid, hydrochinone, and metol, and acts upon the silver bromide in proportion to the intensity of the light falling on the different parts of the plate. The silver bromide is reduced to metallic silver and thus the image is formed. As soon as the image has acquired sufficient density, the plate is washed and placed in a solution of sodium thiosulfate (hypo), which removes all the silver bromide that the light and developing solution have not affected. Since no further change can take place on the plate, the image is said to be " fixed " and the " negative " is made.

b. Action of light on silver bromide. This experiment should be done in a darkened room or in test tubes wrapped in dark paper.

Add 5 cc. of silver nitrate solution to 5 cc. of potassium bromide solution. *Describe the precipitate and the equation for its formation.* Pour a little of the precipitate on a filter paper and place it in the direct sunlight for a few minutes. *Examine it after a few minutes.*

Expose the precipitate remaining in the test tube to a bright light for a few seconds and then add 5 cc. of a developing solution. Allow the action to continue for 2–3 minutes. *Describe any changes occurring in the precipitate.* Then add 5 cc. of a saturated solution of sodium thiosulfate (hypo) and shake the mixture. *What is the effect of adding the thiosulfate?*

c. Silver bromide, not exposed to light. Add 5 cc. of silver nitrate solution to 5 cc. of potassium bromide solution, but without exposing the silver bromide formed to light at all. Then add 5 cc. of the developer. Keep it in the dark for a few minutes. *What is the result?* Add 5 cc. of the " hypo " solution. *Compare the result with that obtained in* (*b*).

d. Printing. Pour a little silver nitrate solution into a shallow dish and float upon it a piece of well-sized paper. Remove the paper and let it dry. Then float it on the surface of a little potassium chloride solution in another dish. Again dry the paper. All this must be done in a darkened room or closet. Place a key or opaque stencil over the paper and expose it to the light for a few minutes. Examine it in a dim light and then immerse the paper in a developing solution for 2 minutes. Rinse it and place it in the " hypo " solution for 10 minutes. Rinse it thoroughly, dry, and mount in your note book.

Optional work. Try to find out the reason for putting sodium carbonate with the photographic developer. Also find the reason for adding a very little potassium bromide to the developing solution. What is the function of sodium sulfite which is often put in the developer? The fixing bath often contains some alum and a little acid. Why?

EXPERIMENT 78

SEPARATION OF SILVER, MERCURY, AND LEAD

Apparatus. — Test tubes and rack. Funnel. Bunsen burner.

Materials. — Solutions of silver, lead, and mercurous nitrates. Hydrochloric acid, concentrated and dilute. Nitric acid, concentrated and dilute. Concentrated and dilute ammonium hydroxide. Solution of potassium chromate. Copper strip. Filter papers. Litmus papers. Unknown solutions.

a. Silver chloride. Add to 5 cc. of silver nitrate ($AgNO_3$) solution an equal volume of water and then add drop by drop dilute hydrochloric acid. Shake between the drops and allow the precipitate to settle until the next drop produces no further precipitation. *What is the precipitate? Write the equation.*

Let the precipitate settle and then pour away the liquid. *What does it contain?* Add to the precipitate a little distilled water and shake the mixture. Allow the precipitate to settle and again pour off the liquid. Now try to dissolve the silver chloride in boiling hot water. *Is silver chloride soluble in hot water?*

Divide the remaining silver chloride into two parts. To one part add dilute ammonium hydroxide. *Is silver chloride soluble in ammonium hydroxide?* Set the other part of the silver chloride in the sunlight and examine it later.

b. Mercurous chloride. To 5 cc. of mercurous nitrate ($HgNO_3$) solution, add 5 cc. of distilled water and then add dilute hydrochloric acid, drop by drop, till the action is complete. *Write the equation.* Wash the precipitate with cold water and then divide into two parts. *Does mercurous chloride dissolve in hot water?* Add to the other portion of the precipitate some concentrated ammonium hydroxide. *How would you test for a soluble mercurous salt?*

c. Lead chloride. Dilute 5 cc. of lead nitrate ($Pb(NO_3)_2$) solution with 5 cc. of distilled water and then add hydrochloric acid, drop by drop, until the precipitation is complete. *Name the precipitate and write the equation.* Wash the precipitate with cold water and boil it with distilled water. *Is lead chloride soluble in hot water?*

Divide the solution into two parts. To one part add a solution of potassium chromate (K_2CrO_4). *What is the precipitate? Write the equation.* Examine the other portion of the solution after it has become cold. *How would you test for a soluble lead salt?*

d. The separation. Make a mixture in one test tube of solutions of silver nitrate, lead nitrate, and mercurous nitrate. Add dilute hydrochloric acid until the precipitation is complete. Filter and wash the precipitated chlorides with a little *cold* water.

To separate the **lead**, wash the precipitate thoroughly with *boiling* water and keep the washings. *Which one of the chlorides will dissolve? Test the filtrate for the presence of this compound.*

Next, to separate the **silver**, wash the precipitate remaining on the filter paper with dilute ammonium hydroxide and keep the washings. *Which chloride is now dissolved? Which remains on the filter?* Add to the filtrate a slight excess of nitric acid (test with litmus paper). *Name the precipitate.*

Finally, to show that the residue left on the filter contains a compound of **mercury**, pour on it a little *aqua regia* (1 cc. of concentrated hydrochloric acid and 0.5 cc. of concentrated nitric acid) and catch the filtrate in a clean test tube. Dilute the solution thus obtained with 5 cc. of water and put into it a small strip of bright copper. After a few minutes, remove the copper, wash it and rub it. *Explain the result.*

e. Unknown. Obtain from the instructor a solution which contains one or more of these metals (silver, mercury, and lead) and analyze the solution. *Report the negative as well as the positive results.*

EXPERIMENT 79

Analysis of a Silver Coin

To determine the percentage of silver in a ten-cent piece.

Apparatus. — Two beakers. Tripod. Asbestos gauze. Bunsen burner. Stirring rod. Funnel. Wash-bottle. Horn pan balance and weights. Ring stand.

Materials. — Dime. Concentrated nitric acid. Dilute hydrochloric acid. Filter papers.

a. Outline. The dime is dissolved in nitric acid, and the metals (silver and copper) are converted into nitrates. The silver, which is precipitated with hydrochloric acid as silver chloride, is filtered off, dried, and weighed. From the weight of the silver chloride, the weight of silver in the coin and the percentage of silver present are calculated.

b. Directions. Weigh to a centigram (0.01 g.) a clean ten-cent piece, place it in a small beaker, and pour in about 20 cc. of nitric acid (diluted with an equal volume of water). As soon as the vigorous action is over, warm the solution gently and add more acid, if necessary, to dissolve the metal. Then dilute the solution by adding 30 cc. of distilled water.

Slowly add hydrochloric acid to the metallic nitrate solution until the precipitation is complete. Keep the solution hot, stirring vigorously, until the precipitate settles readily and leaves the liquid perfectly clear. To prove that the precipitation is complete, add another drop of hydrochloric acid. *How does this show that the precipitation is complete?*

Make ready in a funnel a *weighed* filter paper. Decant the clear liquid down a rod through the filter. Transfer *all* the precipitate to the filter. In order to get out the last particles, use a glass rod and a stream from the wash-bottle (Fig. 100). Wash the precipitate several times with distilled water and then set it aside to dry on the paper. It is well to leave the precipitate in a drying oven where it will become thoroughly dry without burning the paper.

Get the weight of the silver chloride formed. From this and the approximate atomic weights (Table facing front cover) *calculate the weight of silver in a dime.* Finally, *compute the per cent of silver in the coin.*

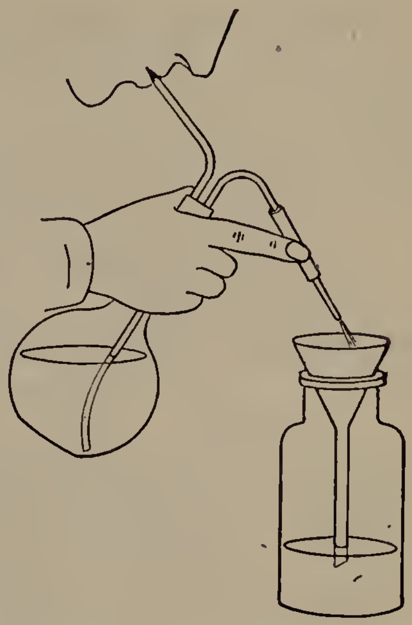

Fig. 100. Washing a precipitate with a wash bottle.

Show all your computations and arrange your data and results in tabular form.

Optional work. To determine the per cent of copper in a dime, keep the filtrate and washings from the silver chloride. Heat the solution nearly to boiling and add slowly, with constant stirring, a clear solution of sodium hydroxide until the precipitation is complete and the liquid gives a decided basic reaction with litmus paper. Cupric hydroxide ($Cu(OH)_2$) is at first formed as a pale blue gelatinous precipitate, but this rapidly turns brown and forms cupric oxide.

Keep the liquid boiling for about 5 minutes and then transfer the precipitate to a filter paper. Dry the filter and precipitate. Burn the filter in a weighed porcelain crucible. Since some of the oxide will be reduced by the burning carbon of the paper, we moisten the residue with one or two drops of concentrated nitric acid. Heat the crucible at first very gently and then strongly to convert the copper nitrate to copper oxide. Cool and weigh. From the weight of copper oxide found, calculate the per cent of copper in a dime.

EXPERIMENT 80

REACTING WEIGHTS OF COPPER AND SILVER

To find the weight of silver which is replaced by 31.8 *grams of copper.*

Apparatus. — Evaporating dish. Graduated cylinder. Horn pan balance and weights. Ring stand. Stirring rod. Funnel. Porcelain crucible. Pipe-stem triangle. Tripod. Bunsen burner. Wash-bottle.

Materials. — Silver nitrate. Copper gauze. Filter papers.

a. Outline. Copper will replace silver from solutions such as silver nitrate and is therefore said to be more active than silver. We may use this fact to determine the combining weight of silver, assuming the combining weight of copper to be 31.8. All we have to do is to weigh the silver replaced by a known weight of copper and then calculate by simple proportion the combining weight of silver.

b. Directions. Dissolve about 4 g. of silver nitrate in 20 cc. of distilled water which is contained in an evaporating dish. Weigh to a centigram (0.01 g.) a piece of bright copper gauze, which ought not to exceed 0.7 g. Immerse the copper gauze completely in the solution and observe that as the copper goes into solution the silver is precipitated. *What evidence have you that the copper is in solution?* Stir the solution frequently with a glass rod until the copper gauze has entirely disappeared.

Then pour the solution through a filter paper in a funnel, leaving the silver in the evaporating dish. Add about 50 cc. of water, stir and pour off the liquid; that is, decant the liquid into the filter paper again, leaving as much of the silver in the dish as possible. In pouring the liquid into the filter, it is well to use the stirring rod and to pour the liquid down along the rod.

Repeat this process of "washing by decantation" twice more; then *transfer all the silver* from the dish to the paper and use the wash-bottle to wash the precipitate. Let the paper and silver drain and then transfer them to a clean porcelain crucible which has been weighed to a centigram. Heat slowly until the paper is dry and then increase the heat until the paper is reduced to white ashes. Cool and weigh again carefully.

Now we have the weight of copper used and the weight of silver deposited. *Calculate the combining weight of silver according to the proportion:*

Wt. of copper : Wt. of silver : : 31.8 : x

Are the combining weights of copper and silver the same as the atomic weights of these metals? Explain.

Optional work. In a similar way determine the combining weight of zinc by precipitating copper from a solution of copper sulfate. In this case wash the copper deposited first with water and then with alcohol on a weighed filter paper. The weight of the filter paper may well be determined by weighing several sheets and then dividing the weight by the number of sheets. The copper on the filter is dried by the air without heating and then reweighed carefully.

Use a little more than 4 g. of copper sulfate crystals for each gram of zinc used.

APPENDIX

PRESSURE OF WATER VAPOR, OR AQUEOUS TENSION

(In millimeters of mercury)

Temperature	Pressure	Temperature	Pressure	Temperature	Pressure
0° C.	4.6 mm.	11° C.	9.8 mm.	22° C.	19.8 mm.
1°	4.9	12°	10.5	23°	21.0
2°	5.3	13°	11.2	24°	22.3
3°	5.7	14°	12.0	25°	23.7
4°	6.1	15°	12.8	26°	25.1
5°	6.5	16°	13.6	27°	26.7
6°	7.0	17°	14.5	28°	28.3
7°	7.5	18°	15.5	29°	29.9
8°	8.0	19°	16.5	30°	31.7
9°	8.6	20°	17.5	50°	92.3
10°	9.2	21°	18.6	100°	760.0

DENSITIES OF IMPORTANT GASES

Approximate weight of 1 liter under standard conditions

Acetylene (C_2H_2)	1.16 grams
Air	1.29 "
Ammonia (NH_3)	0.77 "
Carbon dioxide (CO_2)	1.98 "
Carbon monoxide (CO)	1.25 "
Chlorine (Cl_2)	3.17 "
Helium (He)	0.18 "
Hydrogen (H_2)	0.09 "
Hydrogen chloride (HCl)	1.64 "
Hydrogen sulfide (H_2S)	1.54 "
Methane (CH_4)	0.72 "
Nitric oxide (NO)	1.34 "
Nitrogen (N_2)	1.25 "
Nitrous oxide (N_2O)	1.98 "
Oxygen (O_2)	1.43 "
Sulfur dioxide (SO_2)	2.93 "

THE METRIC SYSTEM

During the last hundred years most of the civilized nations of the world, except Great Britain and the United States, have adopted the **metric system**, and even in these countries most of the scientific work is done in the metric system. In this system of weights and measures the relation of the units is expressed in multiples of **ten.** Fortunately our system of money is made on the decimal plan, that is, in multiples of ten.

Some important units of length. The unit is the **meter** which is equal to 39.37 inches.

1 meter (m.) = 100 centimeters (cm.).
1 centimeter = 10 millimeters (mm.).
1 kilometer (km.) = 1000 meters.
2.54 centimeters = 1 inch.

The chemist needs to become familiar with the centimeter and millimeter. Thus the barometer may read 760 mm. or 76 cm. or about 30 inches.

Units of volume or **capacity.** The unit of volume, that is most extensively used in the laboratory, is the volume of a cube of which the edge is 1 centimeter. This is called a **cubic centimeter** (cc.).

1 liter (l.) = 1000 cubic centimeters (cc.).
1 liter = 1.06 quarts.

Thus a 500 cc. flask holds half a liter or about a pint (16 ounces of water).

Units of Weight. The unit of weight is the **gram** which is approximately the weight of one cubic centimeter of water.

1 gram (g.) = 1000 milligrams (mg.).
1 gram = 100 centigrams (cg.).
1 kilogram (kg.) = 1000 grams.
1 kilogram = 2.2 pounds.
1 ounce (avoirdupois) = 28.35 grams.

The chemist usually expresses weights in grams and decimal fractions thereof, thus a crucible may weigh 12.53 g. Every student in chemistry needs to become very familiar with **centimeters, cubic centimeters,** and **grams.**

TABLE OF SOLUBILITIES

S, soluble in water. P, slightly soluble in water.
I, insoluble in water. Ia, insoluble in water and dilute acids.

	ACETATE	BROMIDE	CARBONATE	CHLORIDE	HYDROXIDE	IODIDE	NITRATE	OXIDE	PHOSPHATE	SULFATE	SULFIDE
Aluminum	S	S	—	S	I	S	S	I	I	S	—
Ammonium	S	S	S	S	S	S	S	—	S	S	S
Barium	S	S	I	S	P	S	S	—	I	Ia	S
Calcium	S	S	I	S	P	S	S	—	I	P	P
Copper	S	S	I	S	I	S	S	I	I	S	Ia
Ferrous (Fe^{++})	S	S	I	S	I	S	S	I	I	S	I
Ferric (Fe^{+++})	—	S	—	S	I	—	S	I	I	S	—
Lead	S	P	I	P	I	I	S	I	I	Ia	Ia
Magnesium	S	S	I	S	I	S	S	I	I	S	I
Manganese	S	S	I	S	I	S	S	I	P	S	I
Mercurous (Hg^{+})	P	I	I	I	—	I	S	I	I	P	Ia
Mercuric (Hg^{++})	S	S	I	S	I	I	S	I	I	S	Ia
Nickel	S	S	I	S	I	S	S	I	I	S	I
Potassium	S	S	S	S	S	S	S	S	S	S	S
Silver	S	Ia	I	Ia	—	Ia	S	I	I	P	Ia
Sodium	S	S	S	S	S	S	S	S	S	S	S
Stannous (Sn^{++})	S	S	—	S	I	S	—	I	I	S	Ia
Stannic (Sn^{++++})	S	S	—	S	I	S	—	I	I	—	Ia
Zinc	S	S	I	S	I	S	S	I	I	S	I

GENERAL RULES FOR SOLUBILITY

(The exceptions to these rules are few and unimportant.)

1. All **sodium, potassium,** and **ammonium** compounds are **soluble** in water.

2. All **nitrates, chlorates,** and **acetates** are **soluble** in water.

3. All **chlorides** are **soluble,** except those of silver, mercury (mercurous), and lead (lead slightly soluble).

4. All **sulfates** are **soluble,** except those of barium, lead, and calcium (calcium slightly soluble). The silver and the mercurous sulfates are only moderately soluble.

5. All **carbonates** are **insoluble,** except those of sodium, potassium, and ammonium.

6. All **oxides** and **hydroxides** are **insoluble,** except those of ammonium, sodium, potassium, and barium (calcium hydroxide slightly soluble).

EXPERIMENTS AND CORRESPONDING CHAPTERS

The Arabic numerals refer to *Black's* Laboratory Experiments, and the Roman numerals to *Black and Conant's* Practical Chemistry.

Experiments	Chapters	Experiments	Chapters
1, 2	I	37, 38	XIX
3, 4	II	39, 40, 41	XX
5, 6	III	42, 43	XXI
7, 8, 9	IV	44, 45, 46, 47	XXII
10	V	48, 49, 50	XXIII
11	VI	51, 52, 53, 54	XXIV
12, 13	VII	55, 56, 57, 58	XXV
14	VIII	59, 60, 61, 62	XXVI
15	IX	63, 64	XXVII
16	X, XI	65, 66	XXVIII
17, 18, 19, 20	XII	67, 68, 69	XXIX
21, 22, 23	XIII	70, 71	XXX
24	XIV	72, 73	XXXI, XXXII
25, 26, 27, 28, 29	XV	74, 75	XXXIII
30, 31, 32	XVI	76, 77, 78, 79	XXXIV
33, 34	XVII	80	XXXV
35, 36	XVIII	Review	XXXVI

LABORATORY SUPPLIES

Apparatus — Individual

Balance,* horn pan, 7½″ beam, sensitive to 0.01 g.
Beakers (3), 100 cc., 200 cc., 300 cc.
Blowpipe, brass, 8″
Bottle, narrow neck, 1000 cc.
Bottles (4), wide mouth, 250 cc.
Brush, test tube
Burner, Bunsen
Clamp, iron, small; for test tubes, burettes, etc.
Crucible, porcelain, with cover, # 0
Crucible, sheet iron with cover, 50 cc.
Dish, crystallizing, 3″ diam.
Dish, evaporating, porcelain, # 1
Dropper, medicine
File, triangular, 15 cm.
Filter papers (25), diam. about 11 cm.
Flask, distilling with side tube, 250 cc.
Flask, Erlenmeyer, 100 cc.
Flask, Erlenmeyer, 200 cc.
Flask, Florence, 300 cc.
Flask, Florence, 500 cc.
Forceps, iron, 3″
Funnel, 60°, 2½″
Gauze, iron with asbestos center, 5″ × 5″
Graduate, cylindrical, 100 cc.
Mortar and pestle, porcelain, about 10 cm.
Pan, agate, shallow, 1 qt.
Pinchcock (Mohr's), medium
Plates, glass (4), 2½″ × 2½″
Platinum wire, # 28, 3″ (or Nichrome wire 6″)
Rack for 12 test tubes
Retort, tubulated, with ground glass stopper, 4 oz.
Rods (2), glass for stirring, 15 cm. long and 3 mm. diam.
Sponge
Spoon, combustion or deflagrating, bowl 1 cm.

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* Some teachers may prefer to use a better balance with agate planes and agate knife edges, in a glass case without rider beam. Under these conditions 2 balances if used in rotation, might serve 10 students.

Stand, iron with 3 rings
Stoppers, rubber with one hole, (2) to fit test tube, (1) to fit ignition tube, (2) to fit combustion tubing, (1) to fit 200 cc. flask
Stoppers, rubber with two holes (1) to fit 500 cc. flask, (2) to fit widemouth bottle, (1) to fit large test tube, (1) to fit 300 cc. flask, (1) to fit 200 cc. flask
Triangle, pipe-stem, size to support # 0 crucible
Tripod, iron, to support dishes over burner
Trough, pneumatic, with shelf or support for bottles
Tube, calcium chloride, 6″
Tube, combustion, 20 cm. long, 15 mm. inside diam.
Tube, ignition (hard glass test tube), 6″ × $\frac{5}{8}$″
Tubes (12), test (soft glass), 6″ × $\frac{3}{4}$″
Tube, test (soft glass), 8″ × 1″
Tube, test, graduated, 30 cc. with 0.5 cc. divisions
Tube, thistle, stem 25 cm. × 6 mm.
Tubing, glass, 200 g., 6 mm. in outside diam.
Tubing, rubber, 2′, $\frac{1}{4}$″ in diam.
Tubing, rubber, 6″, $\frac{3}{16}$″ in diam.
Watch glasses (3), 2$\frac{1}{2}$″ in diam.
Weights,* good grade, 50 g. to 0.01 g., fractional weights in divided compartments under glass cover, all in covered wooden box
Wing-top for Bunsen burner

Apparatus — General. (*For 10 students*)

Note. This list does not include certain apparatus used in the Optional Work.

2 Balances, trip scales with rider (0–10 g.) and weights 10 g. to 500 g.
1 Barometer
1 lb. Beads, glass, small
2 Bottles, acid, about 2$\frac{1}{2}$ liters
4 Burettes, 50 cc. graduated to 0.1 cc.
6 Cells, dry
2 pkg. Cigar lighters, wood splints
10 Cobalt glass plates, double thickness, 2″ × 2″
5 Condensers, Liebig, 15″, with large clamps for ring stand and rubber tubing for connections
1 set Cork-borers (set of 6)
100 Corks, best grade, long, assorted sizes (# 7, 8, 9, 10, 12)
5 Clamps, Hoffman's screw
5 Crucibles, Hessian (sand), 4″ deep

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* See footnote in regard to balances.

1 Cylinder, graduated, 200 cc.
1 Cylinder, graduated, 500 cc.
2 Files, round, in wood handles
1 First aid cabinet with emergency supplies
1 Glass cutter
1 Hydrometer for light liquids
1 doz. Jars, pint fruit
1 Kipp's generator, 1 qt.
2 Magnifying lenses (Coddington)
1 Microscope, compound (1″ eyepiece, $\frac{2}{3}''$ and $\frac{1}{6}''$ objectives)
5 Pans, iron, 5″ shallow form "sand bath"
5 Pipettes, 10 cc.
5 Plates, glass, 4″ × 4″
1 Pliers, side cutting
5 Sets — platinum electrodes for electrical conductivity (Exp. 30) with lamps to suit electric current available
1 doz. Rules, wood, metric, 30 cm.
5 sheets Sandpaper # 1
2 Shears, metal cutting
1 doz. Spoons, iron (tinned)
1 pkg. Tapers, wax
5 Thermometers, graduated from − 10° to + 150° C.
5 Waterbaths, copper, 5″

Materials except for Optional Work. (*For 10 Students*)

1 lb. Acid, acetic (30%) c. p.
1 lb. Acid, formic (50%)
12 lb. Acid, hydrochloric, c. p. (density 1.19)
7 lb. Acid, nitric, c. p. (density 1.42)
8 oz. Acid, oxalic, cryst., c. p.
18 lb. Acid, sulfuric, c. p. (density 1.84)
2 qt. Alcohol, ethyl (95%)
1 lb. Alum, potassium and aluminum sulfate
8 oz. Aluminum sulfate, cryst.
1 lb. Ammonium chloride, c. p.
10 lb. Ammonium hydroxide, c. p. (density 0.90)
1 oz. Ammonium molybdate, cryst., c. p.
8 oz. Ammonium nitrate, cryst., c. p.
2 oz. Ammonium oxalate, cryst., c. p.
1 lb. Ammonium sulfide solution
1 oz. Antimony, powdered
8 oz. Barium chloride, cryst., c. p.

1 lb. Bleaching powder (chloride of lime)
1 lb. Boneblack
1 lb. Borax powder
1 oz. Bromine
1 oz. Cadmium nitrate, c. p.
5 lb. Calcium carbonate (marble chips)
1 lb. Calcium carbonate (precipitated chalk)
2 lb. Calcium chloride (fused or granular)
8 oz. Calcium chloride, cryst., c. p.
2 lb. Calcium oxide (quick lime) in tin can
1 lb. Calcium sulfate, gypsum
5 lb. Calcium sulfate, plaster of Paris
2 lb. Carbon disulfide (commercial)
1 lb. Carbon tetrachloride (commercial)
12 Charcoal blocks for blowpiping
4 oz. Charcoal, wood (powdered)
4 oz. Chromium sulfate, dry, pure
1 oz. Cobalt chloride, c. p.
1 oz. Cobalt nitrate, c. p.
1 oz. Cochineal
32 sq. in. Copper gauze, 80 mesh
8 oz. Copper sheet, $\frac{1}{64}''$ thick
2 lb. Copper turnings, clean, fine
1 lb. spool Copper wire, # 18
1 lb. spool Copper wire, # 24
1 lb. spool Copper wire, # 30
1 lb. Copper chloride, c. p.
8 oz. Copper oxide, powdered, c. p.
8 oz. Copper oxide, wire form
1 oz. Copper sulfate, anhydrous, c. p.
1 lb. Copper sulfate, cryst., c. p.
4 oz. Cotton, absorbent
1 lb. Ether, sulfuric
1 pkg. Fehling's solution tablets
1 lb. Formaldehyde (37%)
10 g. Fuchsine (Rosaniline)
1 oz. Glass wool, fine threads
1 lb. Glucose (sirup)
8 oz. Glycerin, white, U. S. P.
1 lb. Hydrogen peroxide, U. S. P.
1 oz. Iodine, resublimed, U. S. P.
4 oz. Iron chloride (ferric), c. p.

[illegible]

1 lb. Iron filings, clean, fine, or steel wool
8 oz. Iron powder reduced by hydrogen
1 lb. Iron pyrite
1 lb. Iron sulfate (ferrous), cryst., c. p.
1 lb. Iron sulfide (ferrous), sticks, for H_2S
1 pkg. Junket tablets
1 lb. Kaolin (china clay)
1 lb. Lead, metal, foil (tea lead)
4 oz. Lead acetate, cryst., c. p.
8 oz. Lead carbonate (white lead)
1 lb. Lead oxide (litharge)
1 lb. Lead nitrate, c. p.
1 oz. Lithium chloride, c. p.
1 oz. Litmus cubes
2 tubes Litmus paper — 100 strips red
2 tubes Litmus paper — 100 strips blue
1 oz. Magnesium, metal, ribbon
8 oz. Magnesium carbonate, powder
4 oz. Magnesium oxide
1 lb. Magnesium sulfate, cryst. (Epsom salts)
2 lb. Manganese dioxide, gran., free from carbon
4 oz. Manganese dioxide, powdered, c. p.
8 oz. Mercuric oxide, red
4 oz. Mercurous nitrate, pure
4 oz. Mercury, metal, redistilled
10 g. Methyl orange
1 pt. Molasses, good quality
4 oz. Nickel sulfate
1 pt. Olive oil
10 g. Phenolphthalein
1 oz. Phosphorus, red, amorphous
4 oz. Phosphorus, white, sticks
4 oz. Potassium bitartrate (cream of tartar)
4 oz. Potassium bromide, granular, pure
4 oz. Potassium carbonate, cryst., c. p.
1 lb. Potassium chlorate, small cryst., c. p.
4 oz. Potassium chloride, c. p.
4 oz. Potassium chromate, cryst., pure
8 oz. Potassium dichromate cryst., c. p.
8 oz. Potassium ferricyanide, cryst.
4 oz. Potassium ferrocyanide, cryst., c. p.
1 lb. Potassium hydroxide, sticks

2 oz. Potassium iodide, cryst., c. p.
1 lb. Potassium nitrate, cryst., c. p.
1 oz. Potassium oxalate
2 oz. Potassium permanganate, pure
8 oz. Potassium-sodium tartrate (Rochelle salt)
1 oz. Potassium sulfocyanate, c. p.
5 lb. Silica (sand)
4 oz. Silver nitrate, cryst., c. p.
1 lb. Soda lime, granular
4 oz. Sodium, metal
1 lb. Sodium bicarbonate (baking soda)
1 lb. Sodium bisulfite, dry, purified
1 lb. Sodium carbonate, cryst., (washing soda)
8 oz. Sodium carbonate, dry, pure
5 lb. Sodium chloride (common salt), fine, white
2 lb. Sodium hydroxide, sticks
1 lb. Sodium nitrate, cryst., c. p.
8 oz. Sodium peroxide, powd., c. p.
8 oz. Sodium phosphate (disodium), cryst., c. p.
1 lb. Sodium silicate solution (water glass), pure
1 lb. Sodium sulfate, cryst.
2 lb. Sodium thiosulfate (" hypo ")
1 lb. Starch, corn
1 lb. Starch, potato
8 oz. Steel wool
4 oz. Strontium chloride, c. p.
2 lb. Sugar, granulated
2 lb. Sulfur, roll
1 pt. Turpentine
2 lb. Zinc, granulated (mossy)
1 lb. Zinc, sheet
8 oz. Zinc sulfate, cryst.

MISCELLANEOUS SUPPLIES. It should be noted that this list does not include various materials which can be readily obtained at home or at the local stores, such as apples, asbestos paper, baking powders, bread, butter, oleomagarine, soft and hard coal, calico, cotton cloth, woolen cloth and yarn, silk, linen, mixed goods, gasolene, gunpowder, iron nails, kerosene, labels, matches, meat, cornmeal, oatmeal, milk, eggs, peanuts, pinks, Portland cement, rubber bands, Castile soap, vinegar, household ammonia, excelsior, wood chips and splints, candles, photographic developing powders, and yeast cakes.

ADDITIONAL APPARATUS AND MATERIALS FOR OPTIONAL WORK

(The numbers in parentheses refer to Experiments.)

Combustion tube, 30–40 cm. long, 15 mm. inside diam. (14)

Gas measuring tube, 100 cc., graduated to ½ cc. (16)

U-tube, 6″, with two 1-hole rubber stoppers to fit, two platinum electrodes (Fig. 52) (30)

Flask, Florence, 25 cc. (36)

Hydrometer, syringe form for battery testing (36)

Parr's peroxide bomb calorimeter (as made by Standard Calorimeter Co., E. Moline, Ill.) (45)

Gasoline still with electric heater as described by *E. W. Dean* in Technical Papers Nos. 166 and 214 of the Bureau of Mines (47)

Babcock milk-testing outfit for butterfat (53)

Spectroscope, Bunsen type as shown in Fig. 89 (58)

Truog's soil acidity testing set (65)

Tin (foil or granular) (1, 2, 39)
Zinc, powdered (3)
Lead dioxide (4, 59, 64)
Sodium chlorate (6)
Calcium, metallic (7)
Ferric oxide, red (9)
Magnesium, powder (14)
Pyrogallic acid, pure (15)
Aluminum, wire (16)
Iron, piano wire (16, 24)
Tin (stannous) chloride, c. p. (34)
Magnesium chloride (34)
Antimony chloride (34)
Ammonium sulfate (37)
Tin (stannic) oxide (46)
Lead oxide, litharge (46)
Methyl alcohol (48)
Barium dioxide, c. p. (64)
Apatite, calcium phosphate (65)
Lead acetate, cryst. (66)
Zinc sulfide (66)
Alizarin, paste, 20% (69)

In addition to this list there are a few articles which can be readily obtained at home or at the neighborhood store.

Printed in the United States of America.

150 cc

°C

21.65 P in inches.

Feb. 15, 1923

43 cc

16°C

29.6 P in inches

Feb. 16, 1923

FOUR-FIGURE LOGARITHMS

N	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9
10	0000	0043	0086	0128	0170	0212	0253	0294	0334	0374	4	8	12	17	21	25	29	33	37
11	0414	0453	0492	0531	0569	0607	0645	0682	0719	0755	4	8	11	15	19	23	26	30	34
12	0792	0828	0864	0899	0934	0969	1004	1038	1072	1106	3	7	10	14	17	21	24	28	31
13	1139	1173	1206	1239	1271	1303	1335	1367	1399	1430	3	6	10	13	16	19	23	26	29
14	1461	1492	1523	1553	1584	1614	1644	1673	1703	1732	3	6	9	12	15	18	21	24	27
15	1761	1790	1818	1847	1875	1903	1931	1959	1987	2014	3	6	8	11	14	17	20	22	25
16	2041	2068	2095	2122	2148	2175	2201	2227	2253	2279	3	5	8	11	13	16	18	21	24
17	2304	2330	2355	2380	2405	2430	2455	2480	2504	2529	2	5	7	10	12	15	17	20	22
18	2553	2577	2601	2625	2648	2672	2695	2718	2742	2765	2	5	7	9	12	14	16	19	21
19	2788	2810	2833	2856	2878	2900	2923	2945	2967	2989	2	4	7	9	11	13	16	18	20
20	3010	3032	3054	3075	3096	3118	3139	3160	3181	3201	2	4	6	8	11	13	15	17	19
21	3222	3243	3263	3284	3304	3324	3345	3365	3385	3404	2	4	6	8	10	12	14	16	18
22	3424	3444	3464	3483	3502	3522	3541	3560	3579	3598	2	4	6	8	10	12	14	15	17
23	3617	3636	3655	3674	3692	3711	3729	3747	3766	3784	2	4	6	7	9	11	13	15	17
24	3802	3820	3838	3856	3874	3892	3909	3927	3945	3962	2	4	5	7	9	11	12	14	16
25	3979	3997	4014	4031	4048	4065	4082	4099	4116	4133	2	3	5	7	9	10	12	14	15
26	4150	4166	4183	4200	4216	4232	4249	4265	4281	4298	2	3	5	7	8	10	11	13	15
27	4314	4330	4346	4362	4378	4393	4409	4425	4440	4456	2	3	5	6	8	9	11	13	14
28	4472	4487	4502	4518	4533	4548	4564	4579	4594	4609	2	3	5	6	8	9	11	12	14
29	4624	4639	4654	4669	4683	4698	4713	4728	4742	4757	1	3	4	6	7	9	10	12	13
30	4771	4786	4800	4814	4829	4843	4857	4871	4886	4900	1	3	4	6	7	9	10	11	13
31	4914	4928	4942	4955	4969	4983	4997	5011	5024	5038	1	3	4	6	7	8	10	11	12
32	5051	5065	5079	5092	5105	5119	5132	5145	5159	5172	1	3	4	5	7	8	9	11	12
33	5185	5198	5211	5224	5237	5250	5263	5276	5289	5302	1	3	4	5	6	8	9	10	12
34	5315	5328	5340	5353	5366	5378	5391	5403	5416	5428	1	3	4	5	6	8	9	10	11
35	5441	5453	5465	5478	5490	5502	5514	5527	5539	5551	1	2	4	5	6	7	9	10	11
36	5563	5575	5587	5599	5611	5623	5635	5647	5658	5670	1	2	4	5	6	7	8	10	11
37	5682	5694	5705	5717	5729	5740	5752	5763	5775	5786	1	2	3	5	6	7	8	9	10
38	5798	5809	5821	5832	5843	5855	5866	5877	5888	5899	1	2	3	5	6	7	8	9	10
39	5911	5922	5933	5944	5955	5966	5977	5988	5999	6010	1	2	3	4	5	7	8	9	10
40	6021	6031	6042	6053	6064	6075	6085	6096	6107	6117	1	2	3	4	5	6	8	9	10
41	6128	6138	6149	6160	6170	6180	6191	6201	6212	6222	1	2	3	4	5	6	7	8	9
42	6232	6243	6253	6263	6274	6284	6294	6304	6314	6325	1	2	3	4	5	6	7	8	9
43	6335	6345	6355	6365	6375	6385	6395	6405	6415	6425	1	2	3	4	5	6	7	8	9
44	6435	6444	6454	6464	6474	6484	6493	6503	6513	6522	1	2	3	4	5	6	7	8	9
45	6532	6542	6551	6561	6571	6580	6590	6599	6609	6618	1	2	3	4	5	6	7	8	9
46	6628	6637	6646	6656	6665	6675	6684	6693	6702	6712	1	2	3	4	5	6	7	7	8
47	6721	6730	6739	6749	6758	6767	6776	6785	6794	6803	1	2	3	4	5	5	6	7	8
48	6812	6821	6830	6839	6848	6857	6866	6875	6884	6893	1	2	3	4	4	5	6	7	8
49	6902	6911	6920	6928	6937	6946	6955	6964	6972	6981	1	2	3	4	4	5	6	7	8
50	6990	6998	7007	7016	7024	7033	7042	7050	7059	7067	1	2	3	3	4	5	6	7	8
51	7076	7084	7093	7101	7110	7118	7126	7135	7143	7152	1	2	3	3	4	5	6	7	8
52	7160	7168	7177	7185	7193	7202	7210	7218	7226	7235	1	2	2	3	4	5	6	7	7
53	7243	7251	7259	7267	7275	7284	7292	7300	7308	7316	1	2	2	3	4	5	6	6	7
54	7324	7332	7340	7348	7356	7364	7372	7380	7388	7396	1	2	2	3	4	5	6	6	7
	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9

FOUR-FIGURE LOGARITHMS — *Continued*

N	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9
55	7404	7412	7419	7427	7435	7443	7451	7459	7466	7474	1	2	2	3	4	5	5	6	7
56	7482	7490	7497	7505	7513	7520	7528	7536	7543	7551	1	2	2	3	4	5	5	6	7
57	7559	7566	7574	7582	7589	7597	7604	7612	7619	7627	1	2	2	3	4	5	5	6	7
58	7634	7642	7649	7657	7664	7672	7679	7686	7694	7701	1	1	2	3	4	4	5	6	7
59	7709	7716	7723	7731	7738	7745	7752	7760	7767	7774	1	1	2	3	4	4	5	6	7
60	7782	7789	7796	7803	7810	7818	7825	7832	7839	7846	1	1	2	3	4	4	5	6	6
61	7853	7860	7868	7875	7882	7889	7896	7903	7910	7917	1	1	2	3	4	4	5	6	6
62	7924	7931	7938	7945	7952	7959	7966	7973	7980	7987	1	1	2	3	3	4	5	6	6
63	7993	8000	8007	8014	8021	8028	8035	8041	8048	8055	1	1	2	3	3	4	5	5	6
64	8062	8069	8075	8082	8089	8096	8102	8109	8116	8122	1	1	2	3	3	4	5	5	6
65	8129	8136	8142	8149	8156	8162	8169	8176	8182	8189	1	1	2	3	3	4	5	5	6
66	8195	8202	8209	8215	8222	8228	8235	8241	8248	8254	1	1	2	3	3	4	5	5	6
67	8261	8267	8274	8280	8287	8293	8299	8306	8312	8319	1	1	2	3	3	4	5	5	6
68	8325	8331	8338	8344	8351	8357	8363	8370	8376	8382	1	1	2	3	3	4	4	5	6
69	8388	8395	8401	8407	8414	8420	8426	8432	8439	8445	1	1	2	2	3	4	4	5	6
70	8451	8457	8463	8470	8476	8482	8488	8494	8500	8506	1	1	2	2	3	4	4	5	6
71	8513	8519	8525	8531	8537	8543	8549	8555	8561	8567	1	1	2	2	3	4	4	5	5
72	8573	8579	8585	8591	8597	8603	8609	8615	8621	8627	1	1	2	2	3	4	4	5	5
73	8633	8639	8645	8651	8657	8663	8669	8675	8681	8686	1	1	2	2	3	4	4	5	5
74	8692	8698	8704	8710	8716	8722	8727	8733	8739	8745	1	1	2	2	3	4	4	5	5
75	8751	8756	8762	8768	8774	8779	8785	8791	8797	8802	1	1	2	2	3	3	4	5	5
76	8808	8814	8820	8825	8831	8837	8842	8848	8854	8859	1	1	2	2	3	3	4	5	5
77	8865	8871	8876	8882	8887	8893	8899	8904	8910	8915	1	1	2	2	3	3	4	4	5
78	8921	8927	8932	8938	8943	8949	8954	8960	8965	8971	1	1	2	2	3	3	4	4	5
79	8976	8982	8987	8993	8998	9004	9009	9015	9020	9025	1	1	2	2	3	3	4	4	5
80	9031	9036	9042	9047	9053	9058	9063	9069	9074	9079	1	1	2	2	3	3	4	4	5
81	9085	9090	9096	9101	9106	9112	9117	9122	9128	9133	1	1	2	2	3	3	4	4	5
82	9138	9143	9149	9154	9159	9165	9170	9175	9180	9186	1	1	2	2	3	3	4	4	5
83	9191	9196	9201	9206	9212	9217	9222	9227	9232	9238	1	1	2	2	3	3	4	4	5
84	9243	9248	9253	9258	9263	9269	9274	9279	9284	9289	1	1	2	2	3	3	4	4	5
85	9294	9299	9304	9309	9315	9320	9325	9330	9335	9340	1	1	2	2	3	3	4	4	5
86	9345	9350	9355	9360	9365	9370	9375	9380	9385	9390	1	1	2	2	3	3	4	4	5
87	9395	9400	9405	9410	9415	9420	9425	9430	9435	9440	0	1	1	2	2	3	3	4	4
88	9445	9450	9455	9460	9465	9469	9474	9479	9484	9489	0	1	1	2	2	3	3	4	4
89	9494	9499	9504	9509	9513	9518	9523	9528	9533	9538	0	1	1	2	2	3	3	4	4
90	9542	9547	9552	9557	9562	9566	9571	9576	9581	9586	0	1	1	2	2	3	3	4	4
91	9590	9595	9600	9605	9609	9614	9619	9624	9628	9633	0	1	1	2	2	3	3	4	4
92	9638	9643	9647	9652	9657	9661	9666	9671	9675	9680	0	1	1	2	2	3	3	4	4
93	9685	9689	9694	9699	9703	9708	9713	9717	9722	9727	0	1	1	2	2	3	3	4	4
94	9731	9736	9741	9745	9750	9754	9759	9763	9768	9773	0	1	1	2	2	3	3	4	4
95	9777	9782	9786	9791	9795	9800	9805	9809	9814	9818	0	1	1	2	2	3	3	4	4
96	9823	9827	9832	9836	9841	9845	9850	9854	9859	9863	0	1	1	2	2	3	3	4	4
97	9868	9872	9877	9881	9886	9890	9894	9899	9903	9908	0	1	1	2	2	3	3	4	4
98	9912	9917	9921	9926	9930	9934	9939	9943	9948	9952	0	1	1	2	2	3	3	4	4
99	9956	9961	9965	9969	9974	9978	9983	9987	9991	9996	0	1	1	2	2	3	3	3	4
	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9